

## Determination of iodine and iodine compounds in marine samples by ICPMS and HPLC-ICPMS

**Hansen, Maiken Sødergreen; Lewandowski, Daniel Jacob; Rasmussen, Rie Romme; Herbst, Birgitte Koch; Sloth, Jens Jørgen**

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**7<sup>th</sup> Nordic Conference  
on  
Plasma Spectrochemistry**

**June 1- 4, 2014  
Loen, Norway**



**Programme and Abstracts**



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## 2 Welcome Letter

Dear colleagues,

On behalf of the Analytical Section of the Norwegian Chemical Society and the Organising Committee it is an honour and pleasure to welcome you to Loen and the 2014 Nordic Conference on Plasma Spectrochemistry. This conference provides both an international and regional forum by which researchers and users have the opportunity to share their knowledge and exchange ideas.

The plenary programme has been planned with a view of furnishing a comprehensive overview of the latest developments in this scientific field. Since some of the world's leading authorities will be present, the conference will hopefully be a stimulating forum for communication across borders and between scientific disciplines.

By contrast to other plasma spectrochemistry meetings, short tutorial courses will be offered to all participants during the four days meeting which may be attractive to participants who can learn about or to be updated on plasma spectrochemistry. This concept is meant to stimulate especially Nordic users of plasma based techniques to learn more about theoretical and practical issues as well as to obtain specialists recommendations for method improvements.

We know that the natural beauty of the area will captivate you, but we also hope that the conference excursions, social events and outdoor farewell dinner may complement the scientific endeavours. We promise you an exciting and memorable conference and stay in Loen!

Yngvar Thomassen

Chairman

Elin Gjengedal

Vice-Chairman

### 3 Organising and Scientific Committee

**Yngvar Thomassen** (Chairman), National Institute of Occupational Health, Oslo and Norwegian University of Life Sciences, Ås, Norway

**Elin Gjengedal** (Vice-Chairman), Norwegian University of Life Sciences, Ås, Norway

**Balazs Berlinger**, National Institute of Occupational Health, Oslo, Norway

**Walter Lund**, University of Oslo, Norway

**Ivar Martinsen**, GE Healthcare, Oslo, Norway

**Arne Åsheim**, (Exhibition Coordinator), Molab AS, Porsgrunn Branch, Norway

**Sverre Omang** (Treasurer), Oslo, Norway

**Oddvar Røyseth**, NIVA, Norwegian Institute of Water Research, Oslo, Norway

**Jens Sloth**, Technical University of Denmark, National Food Institute, Denmark

### 4 General Information

#### **Conference Desk**

The conference desk is situated in the conference foyer of Hotel Alexandra. It will operate as follows:

Saturday	May 31	16:00 - 18:00
Sunday	June 1	07:30 - 08:15
Monday	June 2	07:30 - 08:00
Tuesday	June 3	07:30 - 08:00
Wednesday	June 4	08:30 - 09:00

Participants are requested to register as soon as possible upon arrival to Hotel Alexandra, Loen.

#### **Conference Venue**

All oral and poster sessions will be held in the various auditoriums of Hotel Alexandra.

Tel: +47 57 87 50 00

Fax: +47 57 87 50 51

Email: [alex@alexandra.no](mailto:alex@alexandra.no)

Homepage: [www.alexandra.no](http://www.alexandra.no)

#### **Meals**

Participants staying at Hotel Alexandra are served breakfast, lunch and dinner (all included in their accommodation package).



## 5 Social Programme

### **Saturday, May 31, 17:00-19:00: Informal Get-together in the Hotel Alexandra Bath & Spa**

All delegates and accompanying persons are invited to enjoy the heated outdoor swimming pool and refreshments.

*This event is kindly sponsored by Holger Hartmann AS.*

### **Sunday, June 1, 14:15: Half-day Excursion to Geiranger**

This tour encompasses a mountain plateau above the tree and snow lines, the summit of Dalsnibba (1746 m), and a sail of 25 km along the spectacular Geirangerfjord.

Price per person: NOK 500 (not included in the Registration Fee).

### **Sunday, June 1, 14:15: Guided Walk to Bøddal Glacier**

On the Bøddal Glacier in Loen Valley you can enjoy a genuine walking experience in beautiful West Norwegian surroundings. The whole trip, rated a moderately difficult hike, takes from five to six hours.

The Bøddal Valley, with the Bøddalseter summer pasture farms and the Bøddal Glacier, is one of the gems of the Jostedalsgreen National Park. Take a rucksack, clothes, food and drink for a half day walk. Good strong mountain boots will be an advantage.

Free of charge.

### **Sunday, June 1, 14:15: Mountain Hiking for Fit Spectroscopists: The Skåla Challenge**

Those who want to challenge the Norwegian mountains are offered a guided walk after lunch to the Klaumann Tower at Skåla Mountain. This is the hardest uphill in entire Norway, approx. 1800 m straight up. Presently there may still be much snow above 1000 meters. Hikers are advised to bring appropriate footwear and clothing for this walk. Free of charge, but we promise; you will sweat!

### **Monday, June 2, 21:30: Bring your own wine**

*This event is kindly sponsored by Agilent Technologies and Matriks AS*

### **Tuesday, June 3, 16:15: Excursion to the Briksdal Glacier with a Conference Outdoor Dinner**

You are invited to visit the heart of Norway - an **Unforgettable** trip to the Briksdal Glacier. Participants can enjoy a walk to view the enormous cascade of ice where you will be able to test the best aquavite in the world; *Gilde Aqua Ultra Plus*, sponsored by Agilent Technologies and Matriks AS.

Afterwards there will be the famous outdoor barbeque at "Kleivane" (weather permitting - otherwise the grill party will be held at Briksdalen Inn).

The well-known Norwegian gourmet chef Frode Aga will be in charge of the kitchen to ensure your best outdoor food experience.

*This event is kindly sponsored by Thermo Scientific and NerliensMeszansky.*

## 6 Scientific Programme

### Oral Presentations

Invited plenary lectures and submitted oral contributions will be 30 and 15 minutes in length, respectively (including discussion).

Video projectors will be provided in all lecture rooms.

### Posters

The posters should be mounted in the early morning on Sunday June 1, in the poster area located next to the lecture room. Materials for poster mounting are available either from the Conference Desk or in the poster mounting area.

Several poster prizes will be awarded sponsored by the journal *Analytical and Bioanalytical Chemistry (ABC)* (two prizes); the Analytical Section of the Norwegian Chemical Society (one prize) and Wiley-VCH (one prize).

### Language

The working language of the conference is English.

## 7 Liability

The Organising Committee declines any responsibility whatsoever for injuries or damages to persons or their property during the Conference.

## 8 Sponsors and Exhibitors

The conference is sponsored by



The exhibition of scientific instrumentation, literature and consumables is located next to the auditorium in the first floor.

The following companies have registered for display and demonstration:

Bruker Daltronics Scandinavia  
CPI International  
Elemental Scientific Inc.  
Holger Hartmann AS  
Houm  
LGC Standards AB  
Agilent Technologies/Matriks AS  
Nu Instruments  
PerkinElmer  
Shimadzu Europa GmbH  
Thermo Scientific/NerliensMeszansky  
Yara Praxair

## **9 Correspondence after the Conference**

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**10 Schedule of Events**

<b>Saturday, May 31, 2014</b>	
16:00 - 18:00	<b>Registration</b>
17:00 - 19:00	<b>Informal Get-together in the Hotel Alexandra Bath &amp; Spa</b> This event is kindly sponsored by Holger Hartmann AS
19:00	<b>Dinner</b>

<b>Sunday, June 1, 2014</b>	
07:30 - 08:30	<b>Registration</b>
08:30 - 08:45	<b>Welcome/Opening Remarks</b>
08:45 - 12:50	<b>Plenary Session I: Progress in Plasma Spectrochemistry</b> Poster Viewing/Exhibition
13:00	<b>Lunch</b>
14:15	<b>Excursion to Geiranger/The Skåla Challenge/Guided Walk to Bødal Glacier</b>
19:00	<b>Dinner</b>

<b>Monday, June 2, 2014</b>				
08:00 - 10:15	<b>Plenary Session II: Speciation Analysis</b> Poster Viewing/Exhibition			
10:35 - 13:05	<b>Plenary Session III: Isotope Ratio Measurements by ICP-MS</b> Poster Viewing/Exhibition			
13:05 - 14:15	<b>Lunch</b>			
14:15 - 15:30	<b>Plenary Session IV: Sample Introduction in Plasma Spectrometry</b>			
<b>Short Course Session 1</b>				
15:30 - 17:15	<b>Short Course A1</b>	<b>Short Course A2</b>	<b>Short Course A3</b>	<b>Short Course A4</b>

<b>Short Course Session 2</b>			
17:45 - 19:30	<b>Short Course B1</b>	<b>Short Course B2</b>	<b>Short Course B3</b>
19:30	<b>Dinner</b>		
21:30	<b>Poster Viewing and Discussions with Wine Tasting (Bring Your Own Wine)</b> This event is sponsored by Matriks AS and Agilent Technologies		

<b>Tuesday, June 3, 2014</b>	
08:00 - 08:15	<b>The Nordic Plasma Torch Award</b>
08:15 - 10:15	<b>Plenary Session V: Progress in Glow Discharge and Laser Ablation Mass Spectrometry Poster Viewing/Exhibition</b>
10:35 - 13:05	<b>Plenary Session VI: Applications of Plasma Spectrochemistry Poster Viewing/Exhibition</b>
13:05 - 14:10	<b>Lunch</b>
<b>Short Course Session 3</b>	
14:10 - 15:55	<b>Short Course C1</b>
	<b>Short Course C2</b>
	<b>Short Course C3</b>
16:15	<b>Excursion to Briksdal Glacier/Conference Outdoor Dinner</b> These events are sponsored by Agilent Technologies, Matriks AS, Thermo Scientific and NerliensMeszansky.

<b>Wednesday, June 4, 2014</b>	
<b>Short Course Session 4</b>	
09:00 - 11:00	<b>Short Course D1</b>
	<b>Short Course D2</b>
	<b>Short Course D3</b>
11:30 - 11:45	<b>Closing Remarks and Farewell</b>
12:00	<b>Lunch</b>

**11 Daily Programme**

Sunday, June 1, 2014

Time	Abstr.
08:30-08:45	<b>Welcome/Opening Remarks</b> <i>Yngvar Thomassen, Conference chairman</i>
<b>Plenary Session I: Progress in Plasma Spectrochemistry</b> <b>Chairman: Jens Sloth</b>	
08:45-09:30	O-1 <b>Distinguished Speaker's Lecture</b> <b>X-ray spectroscopy of astrophysical plasmas</b> Richard E. Griffiths, Department of Physics & Astronomy, University of Hawaii , Hilo, USA
09:30-10:00	O-2 <b>Recent advances in mass cytometry</b> <u>Dmitry R. Bandura</u> , Alexander Loboda, Scott D. Tanner, Vladimir I. Baranov and Olga I. Ornatsky, Fluidigm Canada, Markham, Ontario, Canada
10:00-10:30	O-3 <b>Field-flow fractionation with atomic and mass spectrometry 2014</b> Atitaya Siripinyanond, Department of Chemistry, Mahidol University, Bangkok, Thailand, and <u>Ramon M. Barnes</u> , University Research Institute for Analytical Chemistry, Hadley, MA, USA
10:30-10:50	<b>Coffee, Exhibition and Poster Viewing</b>
10:50-11:20	O-4 <b>New ways to recognize and correct for matrix interferences in plasma based atomic spectrometry</b> <u>Gary M. Hieftje</u> , George Chan, Yan Cheung and Andrew J. Schwartz, Indiana University, Bloomington, IN, USA
11:20-11:35	O-5 <b>Optimization of the ICP-MS interface cone geometry by means of the direct Simulation Monte Carlo method and ion beam modeling</b> <u>Niko Kivel</u> <sup>1</sup> , Heiko-Dirk Potthast <sup>1</sup> , Ines Günther-Leopold <sup>1</sup> , Frank Vanhaecke <sup>2</sup> and Detlef Günther <sup>3</sup> , <sup>1</sup> Paul Scherrer Institute, Nuclear Energy and Safety, Villigen PSI, Switzerland, <sup>2</sup> Ghent University, Department of Analytical Chemistry, Ghent, Belgium, <sup>3</sup> ETH Zürich, Laboratory of Inorganic Chemistry, Zürich, Switzerland
11:35-11:50	O-6 <b>Analysis of nanoparticles using the ICAP-Q-ICP-MS</b> Daniel Kutscher, Shona McSheehy-Ducos, Torsten Lindemann, Thermo Fisher Scientific (Bremen), Germany; <u>Mikael Axelsson</u> , Thermo Fisher Scientific, Hägersten, Sweden; Jörg Bettmer, University of Oviedo, Department for Physical and Analytical Chemistry, Spain; Erik H. Larsen, Technical University of Denmark, National Food Institute, Søborg, Denmark;
11:50-12:05	O-7 <b>Bioanalytical ICP-MS and LC-ICP-MS: new technologies applied during the development and evaluation of new pharmaceutical compounds</b> Jaap Wieling, QPS Netherlands BV, Groningen, The Netherlands
12:05-12:35	O-8 <b>Molybdenum in beans: Ally or enemy of the consumer</b> <u>Katarzyna Wrobel</u> , Kazimierz Wrobel, Alma Rosa Corrales Escobosa Chemistry Department, University of Guanajuato, 36000 Guanajuato, Mexico

**Sunday, June 1, 2014 cont.**

<b>Time</b>	<b>Abstr.</b>
12:35- 12:50	O-9 <b>Development of a dual concentric injector (DCI) ICP torch coupled to a novel, micro cell-in-cell laser ablation chamber for high speed, high efficiency transport of a laser induced aerosols.</b> <i>David N. Douglas<sup>a, b</sup>, Amy J. Managh<sup>a</sup>, Grant Craig<sup>a</sup>, Helen J. Reid<sup>a</sup> &amp; Barry L. Sharp<sup>a</sup></i> <i><sup>a</sup>Centre for Analytical Science, Department of Chemistry, School of Science, Loughborough University, Loughborough, Leicestershire, LE11 3TU, UK. <sup>b</sup>Inorganic Analysis, NMI Operations, Science and Innovation, LGC Group, Queens Rd, Teddington, Middlesex, TW11 0LY, UK</i>
13:00- 14:00	<b>Lunch</b>
14:15-	<b>Excursion to Geiranger/The Skåla Challenge/Guided Walk to Bødal Glacier</b>
19:00-	<b>Dinner</b>

**Monday, June 2, 2014****Plenary Session II: Speciation Analysis****Chairman: Andreas Prange**

<b>Time</b>	<b>Abstr.</b>
08:00- 08:30	O-10 <b>Metal complexes speciation assisted by ICP-MS: Moving frontiers</b> <i>Laurent Querdane</i> and Ryszard Lobinski, Laboratory for Analytical Bioinorganic and Environmental Chemistry, National Research Council of France (CNRS) / University of Pau, France
08:30- 09:00	O-11 <b>Organic and inorganic selenium species in strongly-reducing waters</b> <i>Dirk Wallschläger</i> , Kelly LeBlanc, Jacqueline London and Matthew Smith, Trent University, Peterborough, Ontario, Canada
09:00- 09:30	O-12 <b>Arsenolipids in marine samples - status and analytical challenges</b> <i>Veronika Sele<sup>1</sup>, Heidi Amlund<sup>1</sup> and Jens S. Sloth<sup>1, 2</sup></i> <sup>1</sup> National Institute of Nutrition and Seafood Research, Bergen, Norway <sup>2</sup> National Food Institute, Technical University of Denmark, Søborg, Denmark
09:30- 10:00	O-13 <b>New possibilities for ultra trace multi element and speciation analysis in environmental samples using ICP-MS-MS</b> <i>Daniel Pröfrock</i> , Helmholtz-Centre-Geesthacht, Institute of Coastal Research, Geesthacht, Germany
10:00- 10:15	O-14 <b>Application of isotopic dilution spectrometry for quantification of hexavalent chromium in contaminated soil samples from Northern Italy</b> <i>Laura Guidotti<sup>1</sup>, Silvia Queipo Abad<sup>2</sup>, Pablo Rodriguez Gonzalez<sup>2</sup>, J. Ignacio García Alonso<sup>2</sup>, Gian Maria Beone<sup>1</sup>,</i> <sup>1</sup> Istituto di Chimica Agraria ed Ambientale, Università Cattolica del Sacro Cuore di Milano, sede di Piacenza, Italy <sup>2</sup> Department of Physical and Analytical Chemistry, University of Oviedo, Spain
10:15- 10:35	<b>Coffee Break/Exhibition/Poster Viewing</b>

Monday, June 2, 2014 cont.

**Plenary Session III: Isotope Ratio Measurements by ICP-MS****Chairman: Klaus Heumann**

Time	Abstr.
10:35-11:05	O-15 <b>Application of isotope ratio analysis in ecosystem research</b> <u>Thomas Prohaska</u> , Johanna Irrgeher, Monika Horsky, Ondrej Hanousek, Anastassiya Tchaikovsky and Andreas Zitek, University of Natural Resources and Life Sciences, Department of Chemistry – VIRIS Laboratory, Tulln, Austria
11:05-11:35	O-16 <b>Isotope analysis at trace and ultra-trace levels in environmental matrices</b> <u>Iliia Rodushkin</u> <sup>a,b</sup> , Nicola Pallavicini <sup>a,b</sup> , Emma Engström <sup>a,b</sup> and Douglas C. Baxter <sup>b</sup> <sup>a</sup> Division of Geosciences, Luleå University of Technology, Luleå, Sweden <sup>b</sup> ALS Laboratory Group, ALS Scandinavia AB, Aurorum 10, Luleå, Sweden
11:35-12:05	O-17 <b>Where does it come from? Pb isotopes and ICP-MS in provenance analysis</b> <u>Tom Andersen</u> and Siri Simonsen, Department of Geosciences, University of Oslo, Norway
12:05-12:35	O-18 <b>On-line isotope dilution for routine multi-elemental analysis using ICP-MS</b> <u>Jose.l. García Alonso</u> <sup>1</sup> , J.A. Rodríguez Castrillón <sup>2</sup> and G. Centineo <sup>2</sup> <sup>1</sup> Department of Physical and Analytical Chemistry, Faculty of Chemistry, University of Oviedo, Spain, <sup>2</sup> Innovative Solutions in Chemistry (ISC-science), Campus “El Cristo”, Oviedo, Spain
12:35-13:05	O-19 <b>International culture projects – measurements of skeletons: The ICP-MS in archaeometry</b> Lilian Skytte, CHART, Department of Physics, Chemistry and Pharmacy University of Southern Denmark, Odense, Denmark
13:05-14:15	Lunch

**Plenary Session IV: Sample Introduction in Plasma Spectrometry****Chairman: Ewa Bulska**

Time	Abstr.
14:15-14:45	O-20 <b>Pneumatic nebulizers: a versatile analytical tool for integrating atomic and molecular mass spectrometry for the analysis of elemental species, metalloproteins and engineered nanoparticles</b> Spiros A. Pergantis, Department of Chemistry, University of Crete, Heraklion, Greece
14:45-15:00	O-21 <b>Organic samples analysis by ICP-MS: Which introduction device?</b> <u>Amélie Leclercq</u> <sup>1</sup> , Anthony Nonell <sup>1</sup> , Carole Bresson <sup>1</sup> , Laurent Vio <sup>1</sup> , Frédéric Chartier <sup>2</sup> <sup>1</sup> CEA, DEN, DANS, DPC, SEARS, LANIE, <sup>2</sup> CEA, DEN, DANS, DPC, Gif-sur-Yvette, France
15:00-15:15	O-22 <b>ICP-MS: advancements due to ever-increasing challenges</b> <u>Meike Hamester</u> <sup>1</sup> , Rene Chemnitzer <sup>1</sup> , Iouri Kalinitchenko <sup>2</sup> and Soren Dalby <sup>3</sup> <sup>1</sup> Bruker Daltonics, Fahrenheitstrasse 4, Germany, <sup>2</sup> Iouri Kalinitchenko, Fremont, CA, USA <sup>3</sup> Bruker Daltonics, Hedehusene, Denmark
15:15-15:30	O-23 <b>NexION FAST FIAS unlimited TDS: Direct injection of high (30%) dissolved solid samples</b> <u>Olve Frederichsen</u> <sup>1</sup> and Paul Watson <sup>2</sup> , <sup>1</sup> PerkinElmer As Norge, Oslo, Norway, <sup>2</sup> Elemental Scientific Inc., Omaha, Nebraska, USA

**Monday, June 2, 2014 cont.****Short Course Session 1**

Time	Abstr.	A1:	A2:	A3:	A4:
15:30-17:15	A1- A4	<b>Introduction to Mass Cytometry</b>  Dmitry R. Bandura	<b>Metrological approaches for QA in ICP-MS</b>  Ewa Bulska	<b>Tools to identify metal-containing molecules: Practical aspects to avoid pitfalls</b>  Laurent Ouerdane	<b>Application of inductively coupled plasma atomic emission (ICP-AES) spectrometry – Part I</b>  Joe Brenner

17:15-17:45

**Coffee Break/Exhibition/Poster Viewing****Short Course Session 2**

Time	Abstr.	B1:	B2:	B3:
17:45-19:30	B1- B3	<b>Isotope dilution mass spectrometry for elemental analysis, speciation and metabolism studies</b>  Jose García Alonso	<b>Uncertainty budget cookbook ready to serve in 2 hours</b>  Thomas Prohaska,	<b>The atmospheric pressure glow discharge: Fundamentals and applications</b>  Steven J. Ray

19:30

**Dinner**

21.00

**Poster Viewing and Discussions with Wine Tasting (Bring Your Own Wine)**

This event is sponsored by Matriks AS and Agilent Technologies



Tuesday, June 3, 2014

08:00-  
08:15 **The Nordic Plasma Torch Award**  
Organising Committee

### Plenary Session V: Progress in Glow Discharge and Laser Ablation Mass Spectrometry

*Chairman: Gary Hieftje*

**Time Abstr.**

08:15- 0-24 **New approaches and applications in glow discharge spectroscopy**  
08:45 Steven J. Ray, Andrew Schwartz, Elise Dennis, Andrew Storey and Gary M. Hieftje,  
Department of Chemistry, Indiana University, Bloomington, Indiana, USA

08:45- 0-25 **Depth profile analysis by glow discharge mass spectrometry after ion**  
09:00 **implantation**  
Marisa Di Sabatino, Chiara Modanese and Lars Arnberg, Department of Materials Science  
and Engineering, Norwegian University of Science and Technology (NTNU), Trondheim,  
Norway

09:00- 0-26 **Noise characteristics of an atmospheric pressure glow discharge with a**  
09:15 **solution cathode under inert gas and air environments**  
Stuart Schroeder and Paul Pastushak, Alberta Innovates Technology Futures, Edmonton,  
Alberta, Canada

09:15- 0-27 **Comparison of fast-flow GD-MS and GD-OES methods for depth profiling of Cr**  
09:30 **conversion coatings on Fe substrate**  
Karol Putyera<sup>1</sup> and Arne Bengtson<sup>2</sup>, <sup>1</sup>Evans Analytical Group, Liverpool, New York, USA,  
<sup>2</sup>Swerea KIMAB, Kista, Sweden

09:30- 0-28 **Requirements and capabilities of LA-ICP-MS for trace elements analysis and**  
10:00 **imaging**  
Olga Borovinskaya, Sabrina Gschwind, Joachim Koch, Bodo Hattendorf, Hao Wang and  
Detlef Günther, ETH Zürich, Laboratory of Inorganic Chemistry, Switzerland

10:00- 0-29 **Improved resolution and precision for elemental imaging of geological samples**  
10:15 **using LA-ICP-MS**  
Rene Chemnitzer<sup>1</sup>, Søren Dalby<sup>2</sup> and Meike Hamester<sup>1</sup>  
<sup>1</sup>Bruker Daltonics, Bremen, Germany, <sup>2</sup>Bruker Daltonics Scandinavia, Solna, Sweden

10:15-  
10:35 **Coffee Break/Exhibition/Poster Viewing**

### Plenary Session VI: Applications of Plasma Spectrochemistry

*Chairman: Freddy Adams*

**Time Abstr.**

10:35- 0-30 **Sample preparation strategies for food and biological samples prior to**  
11:05 **nanoparticle detection and imaging**  
Erik H. Larsen and Katrin Loeschner, Technical University of Denmark, National Food  
Institute, Søborg, Denmark

## Tuesday, June 3, 2014 cont.

Time	Abstr.	
11:05- 11:35	O-31	<b>Pushing the limits of single particle ICP-MS for discriminating and sizing small nanoparticles in environmental samples</b> Martin Hassellöv, <u>Jani Tuoriniemi</u> and Geert Cornelis, Department of Chemistry and Molecular Biology, University of Gothenburg, Gothenburg, Sweden
11:35- 11:50	O-32	<b>Handling the high matrix samples with ease using new ICP-MS</b> Uwe Noetzel, Agilent Technologies, Waldbronn, Germany
11:50- 12:20	O-33	<b>Plasma based techniques for biological and clinical applications</b> Ewa Bulska, Faculty of Chemistry, Biological and Chemical Research Center, University of Warsaw, Poland
12:20- 12:35	O-34	<b>ICP based methods for the analysis of energy storage materials</b> <u>Sascha Nowak</u> , Björn Hoffmann, Britta Vortmann, Jennifer Menzel, Timo Schwieters, Martin Winter, University of Münster, MEET Battery Research Center, Germany
12:35- 12:50	O-35	<b>Determination of heavy metals in the food chain using ICP-OES spectrometry</b> <u>Uwe Oppermann</u> <sup>1</sup> , Jan Knoop <sup>1</sup> , Remko Roolvink <sup>1</sup> and Albert van Oyen <sup>2</sup> <sup>1</sup> Shimadzu Europa GmbH, Duisburg, Germany, <sup>2</sup> Carat GmbH, Bocholt, Germany
12:50- 13:05	O-36	<b>Improved methodology for calibrating linear responses in routine analysis</b> Arngrímur Thorlacius, Agricultural University of Iceland, Borgarnes, Iceland
13:05- 14:10		<b>Lunch</b>

**Short Course Session 3**

Time	Abstr.	C1:	C2:	C3:
14:10- 15:55	C1- C3	<b>Sample preparation in trace element analysis</b>  Dirk Wallschläger	<b>ICP-MS applications in epigenetic studies</b>  Katarzyna Wrobel	<b>Continuous fast scanning single particle ICP-MS for detection and distribution determinations: Theory and applications</b>  Fadi Abou Shakra

16:15- **Excursion to Briksdal Glacier/Conference Outdoor Dinner**  
These events are sponsored by Agilent Technologies, Matriks AS, Thermo Scientific and NerliensMeszansky.

Wednesday, June 4, 2014

**Short Course Session 4**

Time	Abstr.	D1:	D2:	D3:
09:00-11:00	D1- D3	<b>Application of inductively coupled plasma atomic emission (ICP-AES) spectrometry – Part II</b>  Joe Brenner	<b>Systematic approach to analytical problem solving</b>  Ramon M. Barnes	<b>Hyphenated techniques: Overview about recent tools and developments for marine environmental speciation analysis</b>  Daniel Pröfrock

11:00-11:30

**Coffee Break**

11:30-11:45

**Closing Remarks and Farewell**

12:00-

**Lunch**

**12 Poster Presentations****Abstr. Sunday, June 1 - Wednesday, June 4, 2014**

- P-1 Boron speciation and accurate boron quantification in metallurgical grade silicon using HPLC-ICP-MS and ICP-OES**  
Patrick Galler<sup>1</sup>, Andrea Raab<sup>2</sup>, Sabine Freitag<sup>2</sup>, Kjell Blandhol<sup>1</sup>, Jörg Feldmann<sup>2</sup>  
<sup>1</sup> Elkem AS, Technology, Central Analytical Laboratory, Fiskaaveien 100, P.O. Box 8040 Vaagsbygd, NO-4675 Kristiansand, Norway. E-mail: patrick.galler@elkem.no  
<sup>2</sup> University of Aberdeen, College of Physical Science, Department of Chemistry, Trace Element Speciation Laboratory (TESLA), Meston Walk, Aberdeen AB24 3UE, Scotland, United Kingdom.
- P-2 Identification of mercury compounds in gypsum samples from a lignite burning thermo power plant**  
Majda Pavlin<sup>1,3</sup>, Matej Sedlar<sup>2,3</sup>, Milena Horvat<sup>1,3</sup>, Radojko Jaćimović<sup>1</sup>, Arkadij Popovič<sup>1</sup>  
<sup>1</sup> Jožef Stefan Institute, Department of Environmental Sciences, Ljubljana, Slovenia  
<sup>2</sup> Esotech, d.d., Velenje, Slovenia  
<sup>3</sup> Jožef Stefan International Postgraduate School, Ljubljana, Slovenia
- P-3 Measurement of copper diffusivity in silicon by glow discharge mass spectrometry**  
Chiara Modanese, Guilherme Gaspar, Lars Arnberg, Marisa Di Sabatino.  
Norwegian University of Science and Technology (NTNU), Alfred Getz v. 2B, N-7491 Trondheim, Norway.
- P-4 Quantification of ten trace elements in foodstuffs by ICP-MS**  
Dubascoux S., Nicolas M., Richoz-Payot J., Poitevin E. & Perring L.  
Nestle Research Center, Analytical Sciences Competence Pillar - Minerals & Imaging Group, P.O. Box 44, CH - 1000 Lausanne 26
- P-5 Cadmium isotope ratio measurements in environmental matrices by MC-ICP-MS**  
Nicola Pallavicini<sup>a,b</sup>, Emma Engström<sup>a,b</sup>, Douglas C. Baxter<sup>b</sup>, and Ilia Rodushkin<sup>a,b</sup>  
<sup>a</sup> Division of Geosciences, Luleå University of Technology, S-971 87 Luleå, Sweden  
<sup>b</sup> ALS Laboratory Group, ALS Scandinavia AB, Aurorum 10, S-977 75 Luleå, Sweden
- P-6 Variability in osmium, lead, zinc and cadmium isotope composition in birch leaves from Sweden**  
Nicola Pallavicini<sup>a,b</sup>, Emma Engström<sup>a,b</sup>, Douglas C. Baxter<sup>b</sup>, and Ilia Rodushkin<sup>a,b</sup>  
<sup>a</sup> Division of Geosciences, Luleå University of Technology, S-971 87 Luleå, Sweden  
<sup>b</sup> ALS Laboratory Group, ALS Scandinavia AB, Aurorum 10, S-977 75 Luleå, Sweden
- P-7 An exploration of sample preparations and detection limits for conductive, semi-conductive and non-conductive materials as pertaining to the Nu Astrum HR-GDMS.**  
Dr DeAnn Barnhart<sup>1</sup>, Glyn Churchill<sup>1</sup>, Andrew Burrows<sup>1</sup>, Bengt Sandgårde<sup>2</sup>  
<sup>1</sup> Nu Instruments Limited, Unit 74, Clywedog Rd S, Wrexham Industrial Estate, Wrexham, Clwyd UK LL13 9XS. <sup>2</sup> Massanalys Spectrometry Nordic AB, Biskopsvagen 9, SE 12352 Farsta, Sweden
- P-8 Determination of essential trace elements of cashew apple powder from Venezuela by inductively coupled plasma mass spectrometry**  
Alfonso Bravo<sup>1\*</sup>, Manuel Amaro López<sup>2</sup>, Jaisel Puentes<sup>1</sup>, Jorly Mejia<sup>1</sup>, Daniel Villalobos<sup>1</sup>, Dairlex Bravo<sup>1</sup>, Dariangelis Bravo<sup>1</sup>, Brinolfo Montilla<sup>3</sup>, Marinela Colina<sup>3</sup>  
<sup>1</sup> Nutrition Research and Development Laboratory, University of Zulia, Venezuela.  
<sup>2</sup> Department of Bromatology and Food Technology. University of Cordoba, Spain.  
<sup>3</sup> Environmental Chemistry Laboratory, Department of Chemistry, University of Zulia, Venezuela. Maracaibo, 4011.

Abstr.

## Poster Presentations cont.

- P-9 Estimation of gas temperature from emission of OH in mercury-containing capillary light sources**  
E. Bogans<sup>1</sup>, Z. Gavare<sup>1</sup>, N. Zorina<sup>1</sup>, A. Svagere<sup>1</sup>, A. Skudra<sup>1</sup>  
<sup>1</sup>Institute of Atomic Physics and Spectroscopy, University of Latvia, Skunu str. 4, Riga, LV-1050, Latvia.
- P-10 Determination of gas temperature using rotational spectra of OH and C2 in thallium-containing light sources**  
Z. Gavare<sup>1</sup>, E. Bogans<sup>1</sup>, M. Zinge<sup>1</sup>, A. Skudra<sup>1</sup>  
<sup>1</sup>Institute of Atomic Physics and Spectroscopy, University of Latvia, Skunu str. 4, Riga, LV-1050, Latvia.
- P-11 Determination of Zn, Cu, Pb and Fe in wine by microwave plasma-atomic emission spectrometer**  
Andrea Carcano<sup>1</sup>, Paolo Bellassi<sup>2</sup>, Gian Maria Beone<sup>2</sup>  
<sup>1</sup>Agilent Technologies, Inc. <sup>2</sup>Istituto di Chimica Agraria ed Ambientale, Università Cattolica del Sacro Cuore di Piacenza
- P-12 Development and validation of a method for determination of 12 elements in breast milk by ICP-MS-QQQ**  
Enger, Ø., Jensen, K.A. & Gjengedal, E.  
Department of Environmental Sciences, Norwegian University of Life Sciences, N-1432 Aas, Norway.
- P-13 Separation of lanthanoides on reverse phase columns modified with various ligands: utilizing ICP-MS as detector**  
Muhammad Ramzan, Dejene Kifle and Grethe Wibetoe  
Department of Chemistry, University of Oslo, P.O.Box 1033 Blindern, N-0315 Oslo, Norway
- P-14 Elimination of molecular interferences when using QP-ICP-MS: determination of fifteen rare earth elements in europium- and ytterbium matrices utilizing ICP-MS and HPLC-ICP-MS**  
Viet Hung Nguyen, Dejene Kifle and Grethe Wibetoe  
Department of Chemistry, University of Oslo, P.O.Box 1033 Blindern, N-0315 Oslo, Norway
- P-15 Investigation of elemental distribution in lithium ion battery components using LA-ICP-MS**  
Björn Hoffmann<sup>1</sup>, Martin Winter<sup>1</sup>, Sascha Nowak<sup>1\*</sup>  
\*(sascha.nowak@uni-muenster.de)  
<sup>1</sup>University of Münster, MEET, Corrensstraße 46, 48149 Germany
- P-16 Determination of lithium and transition metals in lithium-ion battery cells by inductively coupled plasma – optical emission spectrometry and inductively coupled plasma – mass spectrometry**  
Britta Vortmann<sup>1</sup>, Björn Hoffmann<sup>1</sup>, Martin Winter<sup>1</sup> and Sascha Nowak<sup>1</sup>  
<sup>1</sup>University of Münster, MEET, Corrensstraße 46, 48149 Münster, Germany
- P-17 Quantification of phosphorous containing degradation products in LiPF<sub>6</sub> based electrolytes with IC/ICP-MS**  
Jennifer Menzel<sup>1\*</sup>, Vadim Kraft<sup>1</sup>, Markus Knepper<sup>2</sup>, Martin Winter<sup>1</sup> und Sascha Nowak<sup>1</sup>  
<sup>1</sup>MEET – Batterieforschungszentrum, Corrensstraße 46, 48149 Münster, Germany  
\*E-Mail: Jennifer.menzel@uni-muenster.de  
<sup>2</sup>Deutsche METROHM GmbH & Co. KG, Technisches Büro Nordrhein-Westfalen, Kaninenberghöhe 8, 45136 Essen, Germany

Abstr.

## Poster Presentations cont.

- P-18 Quantitative determination of aging related changes in the lithium distribution in LFP electrodes.**  
Timo Schwieters, Bjoern Hoffmann, Martin Winter and Sascha Nowak  
University of Muenster, Institute of Physical Chemistry, D-48149 Muenster, Germany
- P-19 Speciation analysis of antimony in diffusive gradients in thin films (DGT) by high performance liquid chromatography - isotopic dilution - inductively coupled plasma mass spectrometer (HPLC-ID-ICP-MS)**  
Fontanella Maria Chiara, Gian Maria Beone, Ilenia Cattani.  
Institute of Agricultural and Environmental Chemistry, Università Cattolica, Via E. Parmense 84, Piacenza.
- P-20 Serum levels of essential minerals in nurse students with smoking habits from the Zulia university, Venezuela**  
Fred de La Hoz<sup>1</sup>, Nataly Zerpa<sup>1</sup>, Daniel Cárdenas<sup>1</sup>, Alfonso Bravo<sup>2</sup>, Dulce Perozo<sup>1</sup>, Dairlex Bravo<sup>1</sup>, Dariangelis Bravo<sup>1</sup>, Brinolfo Montilla<sup>3</sup>  
<sup>1</sup>School de Nursing, Faculty of Medicine, University of Zulia, Venezuela.  
<sup>2</sup>Nutrition Research and Development Laboratory, University of Zulia, Venezuela.  
<sup>3</sup>Environmental Chemistry Laboratory, Department of Chemistry, University of Zulia, Venezuela. Maracaibo, 4011.
- P-21 Analytically monitoring the effect of fracturing activity**  
<sup>1</sup>Shona McSheehy Ducos, <sup>1</sup>Rob Henry, <sup>1</sup>Jianfeng Cui, <sup>1</sup>Julian D. Wills, <sup>2</sup>Matthew Cassap, <sup>3</sup>Richard Jack, <sup>4</sup>Mikael Axelsson;  
<sup>1</sup>Thermo Fisher Scientific, Hanna-Kunath-Straße 11, 28199 Bremen, Germany; <sup>2</sup>Thermo Fisher Scientific, SOLAAR House, 19 Mercers Road, Cambridge, UK; <sup>3</sup>Thermo Fisher Scientific, 1214 Oakmead Parkway, Building 10, Sunnyvale, CA, USA; <sup>4</sup> Thermo Fisher Scientific, Telefonvägen 30, 126 26 Hagersten, Sweden
- P-22 Dissolution studies of sparingly soluble ThO<sub>2</sub> with HR-ICP-MS**  
Emmi Myllykylä<sup>1</sup>, Tiina Lavonen<sup>1</sup> and Kajja Ollila<sup>1</sup>  
<sup>1</sup>VTT Technical Research Centre of Finland, P.O. Box 1000, FI-02044 VTT)
- P-23 Effects of some heavy metals on the Rhizobacterium Azospirillum Brasilense: spectrochemical analyses of metal uptake and metabolic responses**  
Alexander A. Kamnev, Anna V. Tugarova  
Institute of Biochemistry and Physiology of Plants and Microorganisms, Russian Academy of Sciences, 13 Prospekt Entuziastov, 410049 Saratov, Russia
- P-24 New DGT adsorbents for dissolved inorganic and organic phosphorus compounds in water (DIP and DOP)**  
Oddvar Røyset<sup>1</sup>, Tomas Adler Blakset<sup>1</sup>, Rolf Vogt<sup>2</sup>, Christian Mohr<sup>2</sup>, Neha Parekh<sup>2</sup>,  
1) Norwegian Institute for Water Research (NIVA), Gaustadalléen 21, N-0349 OSLO, +47 90149541, [oddvar.roeyset@niva.no](mailto:oddvar.roeyset@niva.no), 2) Department of Chemistry, University of Oslo, 0315 OSLO, Norway.
- P-25 DGT-probes applied for determination of metal distribution and metal exchange across the sediment-water interface in Sørfjorden, Norway.**  
Morten Schaanning, Anders Ruus, Sigurd Øxnevad and Oddvar Røyset,  
Norwegian Institute for Water Research (NIVA), Gaustadalléen 21, N-0349 OSLO, Norway, [www.niva.no](http://www.niva.no), +47 99230782, [mts@niva.no](mailto:mts@niva.no)
- P-26 Benefits of transitioning from FAAS to the Agilent 4200 MP-AES**  
Uwe Noetzel  
Agilent Technologies, Hewlett-Packard-Strasse 8, D-76337 Waldbronn, Germany



**Abstr.**

**Poster Presentations cont.**

**P-27 Single particle analysis of nanomaterials with Agilent 7900 ICP-MS**

Uwe Noetzel

Agilent Technologies, Hewlett-Packard-Strasse 8, D-76337 Waldbronn, Germany

**P-28 Bioavailability of cadmium from linseed and cocoa**

Max Hansen, Rie R. Rasmussen and Jens J. Sloth

National Food Institute (DTU Food), Technical University of Denmark

**P-29 Determination of iodine and iodine compounds in marine samples by ICP-MS and HPLC-ICP-MS**

Maiken S. Hansen, Daniel J. Lewandowski, Rie R. Rasmussen, Birgitte K. Herbst and Jens J. Sloth

National Food Institute (DTU Food), Division of Food Chemistry – Technical University of Denmark, Søborg, Denmark

**P-30 Strategies for the measurement of lead isotope ratios in radiogenic minerals by laser ablation multicollector ICP-MS**

A. Reguera Galan, M. Moldovan and J.I. Garcia Alonso

Department of Physical and Analytical Chemistry, Faculty of Chemistry, University of Oviedo. Julian Claveria 8, 33006 Oviedo, Spain.

## 12 Oral Abstracts

**(O-1)**

### **X-RAY SPECTROSCOPY OF ASTROPHYSICAL PLASMAS**

*Richard E. Griffiths*

*Dept. of Physics & Astronomy, University of Hawaii , Hilo  
Emeritus Professor, Carnegie Mellon University, Pittsburgh, PA*

X-ray spectroscopy now provides a powerful technique for the investigation of X-ray emitting plasmas in a wide variety of cosmic X-ray sources.

Following a brief review of the techniques which have been under development since the 1960's, we will show how discrete X-ray line diagnostics are now used to provide unique and unambiguous measurements of temperatures, densities, excitation conditions, ionization balance and elemental abundances in these sources, which range from supernova remnants and binary stars in our own Milky Way Galaxy, to massive black holes in the early universe and collisions of clusters of galaxies. Results from the two major extant X-ray observatories, ESA's XMM-Newton and NASA's Chandra X-ray Observatory, will be reviewed.

**(O-2)**

**RECENT ADVANCES IN MASS CYTOMETRY**

*Dmitry R. Bandura, Alexander Loboda, Scott D. Tanner, Vladimir I. Baranov and Olga I. Ornatsky*

*Fluidigm Canada, Markham, Ontario, Canada*

*dmitry.bandura@fluidigm.com*

Mass Cytometry uses specific, fit for the purpose implementations of the inductively coupled plasma ion source, time-of-flight analyzer, dual digital/analog ion signal processing and a cell introduction system.

The overall efficiency of a Mass Cytometer system in quantifying antigens in real time single cell assays depends on several factors. First, processes of aerosol generation, vaporization, atomization and ionization of single cells define how quantitatively the single-cell induced ion cloud sampled through the plasma-vacuum interface represents the composition of the single cell. Second, ion transport through the interface, the ion optical path and the time-of-flight analyzer define not only how few molecules per cell can be detected (dependent on the labelling efficiency), but also how readily the bright and the dim labels can be distinguished while retaining high specificity and high multiplexing capability of the assay. Third, the ion signal handling, occurring on the nano-second scale and including ion detection, signal pre-amplification, digitization and processing, defines the dynamic range of a single cell assay. It also enables sampling of cell-induced transients on the microsecond scale to provide additional gating information to resolve doublets and debris from true single cell events.

We will discuss advances in the hardware related to each of the above topics, and will present data for leukemia cell lines to demonstrate the achievement of the goals.

**(O-3)****FIELD-FLOW FRACTIONATION WITH ATOMIC AND MASS SPECTROMETRY  
2014**

*Atitaya Siripinyanond, Department of Chemistry, Mahidol University, Bangkok 10400, Thailand, and  
Ramon M. Barnes,*

*University Research Institute for Analytical Chemistry, PO Box 666, Hadley, MA 01035, USA,  
barnes@chemistry.umass.edu*

The detection and characterization of natural and engineered nanoparticles and macromolecules have received considerable attention recently as many household, medical, and commercial products incorporate nanomaterials. Nanomaterials are applied in numerous fields owing to their size, surface characteristics, and other unique chemical and physical properties. Nanotechnology is present widely, and the incorporation of nanoparticles is common in many applications such as nanotubes for textiles, nanotechnology for sunscreens, and nanoparticles in body care products such as shower gels and body lotions. Metals and metal oxides are among the mostly frequently employed nanoparticles. The increasing production and dissemination of engineered nanoparticles could lead to potential health and environmental risks. Field-flow fractionation (FFF) using flow (FFFF) or sedimentation (SdFFFF) fields provide separations related to particle size and/or molecular weight [1-7]. Combined on-line with inductively coupled plasma (ICP) spectrometry detection, the elemental and isotopic distributions in separated fractions can be identified and quantified. FFF also can be combined off-line with electrothermal vaporization atomic absorption, emission, or mass spectrometry. This review will describe recent instrumental developments and applications of FFF with atomic and mass spectrometric detection especially for natural and synthetic nanoparticle characterization.

[1] S.K.R. Williams, et al., *Field-Flow Fractionation: Addressing the Nano Challenge*, *Anal. Chem.*, **83**, 634-642 (2011).

[2] S. Dubascoux, et al., *Field-flow Fractionation and Inductively Coupled Plasma Mass Spectrometer Coupling: History, Development and Applications*, *J. Anal. At. Spectrom.*, **25**, 613-623 (2010).

[3] R.N. Qureshi and W.T. Kok, *Application of Flow Field-Flow Fractionation for the Characterization of Macromolecules of Biological Interest: A Review*, *Anal. Bioanal. Chem.*, **399**, 1401-1411 (2011).

[4] F. von der Kammer, S. Legros, T. Hofmann, E.H. Larsen, K. Loeschner, *Separation and Characterization of Nanoparticles in Complex Food and Environmental Samples by Field-Flow Fractionation*, *TrAC Trends in Analytical Chemistry*, **30**(3), 425-436 (2011).

[5] M. Baalousha, B. Stolpe, J.R. Lead, *Flow field-flow fractionation for the analysis and characterization of natural colloids and manufactured nanoparticles in environmental systems: A critical review*, *Journal of Chromatography A*, **1218**(27), 4078-4103 (2011).

[6] Karl-Gustav Wahlund, *Flow field-flow fractionation: Critical overview*. *Journal of Chromatography A*, **1287**, 97-112 (2013)

[7] S. Kim R. Williams, Karin D. Caldwell, (Eds.) *Field-flow Fractionation in Biopolymer Analysis*, Springer, (2012).

**(O-4)****NEW WAYS TO RECOGNIZE AND CORRECT FOR MATRIX INTERFERENCES IN PLASMA-BASED ATOMIC SPECTROMETRY***Gary M. Hieftje, George Chan, Yan Cheung, and Andrew J. Schwartz**Department of Chemistry, 800 East Kirkwood Ave., Bloomington IN 47405 USA**e-mail: Hieftje@indiana.edu*

We have been engaged in devising strategies for understanding and alleviating matrix interferences in ICP emission spectrometry. Such interferences arise when a given analyte concentration in standards and samples does not produce the same signal level. These errors are generally caused by differences in the efficiency with which atoms and ions are generated and excited in the plasma and have been the subject of research in our laboratory for some time. Recently, we have begun exploring end-on observation of ICP emission; in this mode, signals are higher and precision is better, but matrix interferences are worse, so recognizing them is even more critical. We have also been applying our error-recognition methods to the analysis of organic samples, where again interferences are known to be worse. The recent effort involves measuring either the spatial distribution of analyte emission or the effect of dilution on the magnitude of interference. Because the ICP is spatially heterogeneous, especially in an end-on observation mode, the magnitude of interference changes from one plasma location to another. Indeed, even with a fixed analyte and interferent concentration, the effect can change from a signal enhancement in one location to a suppression in another. The interference-recognition method is then straightforward: a calibration curve (emission intensity vs. concentration) is created at a number of spatial locations in the ICP by using a series of solutions of known concentration. The emission signal from an unknown sample is then obtained at each of those locations and its apparent concentration determined at each location by reference to the corresponding calibration curve. If the sample behaves in the same way as the standards, the determined concentrations will be the same for all spatial locations. If the determined concentrations differ, a matrix interference clearly exists. The dilution-based method is similarly straightforward; because different analyte elements are affected dissimilarly by a matrix interferent, the degree of matrix-induced signal enhancement or suppression changes from one element to another during dilution. Ratioing two analyte signals as dilution proceeds then yields a constant value in the absence of an interferent but a changing value when an interference is present.

**(O-5)****OPTIMIZATION OF THE ICP-MS INTERFACE CONE GEOMETRY BY MEANS OF THE DIRECT SIMULATION MONTE CARLO METHOD AND ION BEAM MODELING.***Niko Kivel<sup>1</sup>, Heiko-Dirk Potthast<sup>1</sup>, Ines Günther-Leopold<sup>1</sup>, Frank Vanhaecke<sup>2</sup> and Detlef Günther<sup>3</sup>**<sup>1</sup>Paul Scherrer Institute, Nuclear Energy and Safety, 5232 Villigen PSI, Switzerland**<sup>2</sup>Ghent University, Department of Analytical Chemistry, Krijgslaan 281 - S12, 9000 Ghent, Belgium**<sup>3</sup>ETH Zürich, Laboratory of Inorganic Chemistry, Vladimir-Prelog-Weg 1-5/10, 8093 Zürich, Switzerland*

The interface between the atmospheric pressure plasma ion source and the high vacuum mass spectrometer is one of the most important parts of an ICP-mass spectrometer. It has impact on the efficiency of the mass transfer into the mass spectrometer, as well as it contributes to the formation of interfering ions and mass discrimination.

This region of the ICP-MS interface was simulated using the Direct Simulation Monte Carlo (DSMC) method with respect to the formation of shock waves, mass transport and mass discrimination. The simulations form the basis for an improvement of the cone design towards higher mass transport with lower mass discrimination. Such optimization is feasible due to the insights gained into the effects and geometrical features causing mass discrimination.

Aside from the overall mass transport phenomena, the impacts of the cone geometry on the electric field in the mass spectrometer have to be accounted for. This is achieved by means of ion beam modeling with respect to the encountered beam currents and expected plasma potential. Earlier studies suggested a wider skimmer base and a shorter tip in order to allow the electric field to penetrate deeper into the skimmer cone. A priori this would improve the acceleration of the extracted ions towards the mass spectrometer and therefore, reduce the impact of space-charge forces.

The presentation will provide a comparison of the modeling results to experimental data for existing cone designs. Additionally first results of the cone geometry optimization will be presented.



**(O-6)****ANALYSIS OF NANOPARTICLES USING THE ICAP Q ICP-MS.**

*Daniel Kutscher, Shona McSheehy-Ducos, Torsten Lindemann, Thermo Fisher Scientific (Bremen), Hanna-Kunath-Str. 11, 28199 Bremen Germany; Mikael Axelsson, Thermo Fisher Scientific,*

*Telefonvägen 30, 126 26 Hägersten, Sweden; Jörg Bettmer, University of Oviedo, Department for Physical and Analytical Chemistry, 33006 Oviedo, Spain; Erik H. Larsen, Technical University of Denmark, National Food Institute, Mørkhøj Bygade 19, 2860 Søborg, Denmark;*

The analysis of Nanoparticles (NPs) has become one of the hot topics in analytical chemistry. However, detailed knowledge about potential risks or hazards is still unavailable. Two approaches have been developed in recent years using ICP-MS as a detection system: hyphenation of an appropriate separation technique like Field-Flow-Fractionation (FFF), or direct analysis using spICP-MS.

The separation of particles and particle mixtures using FFF is based on the differing mobilities of different particle sizes in a laminar liquid flow. FFF is compatible for particle sizes in the low nm to low  $\mu\text{m}$  range and is thus perfectly suited for the separation of different NPs. Combined with other detection systems such as dynamic light scattering, it can provide also additional information about the particles of interest, like shape.

In comparison, spICP-MS is able to analyze NPs directly based on the signal intensity of single particle events in the plasma which are directly proportional to the size of the NPs. This direct approach greatly simplifies the experimental set-up. Using ICP-MS instrumentation with the very highest elemental sensitivity, NPs with diameters in the low nm range can be analyzed.

In this presentation, the basic terms of both techniques will be presented. The key benefits and drawbacks of each technique are illustrated in results from samples contain NPs of different structure and size. The challenges that remain for the characterization of NPs in real samples such as environmental matrices or foods will be briefly discussed.

**(O-7)**

**BIOANALYTICAL ICP-MS AND LC-ICP-MS: NEW TECHNOLOGIES APPLIED DURING THE DEVELOPMENT AND EVALUATION OF NEW PHARMACEUTICAL COMPOUNDS**

*Jaap Wieling, QPS Netherlands BV, Petrus Campersingel 123, 9713 AG Groningen, The Netherlands, jaap.wieling@qps.com*

In today's bioanalysis, the vast majority of bioanalytical methods for PK and PD purposes are based on LC-MS/MS and immunochemical principles. However, an increasing group of compounds have molecular characteristics that open opportunities for the application of elemental detection by ICP-MS. This technique offers orthogonal properties to molecular detection and binding properties. Hence this may also provide superior capabilities in specific cases. This is also true for bioanalytical challenges for which bioanalysts would normally chose LC-MS/MS based methods, e.g. protein biomarkers and proteomics; in these domains elemental tagging provides these molecules with excellent characteristics for ICP-MS detection.

Here, the analytical potential, the various modes of operation and the challenges of the application of ICP-MS in drug development applications are given, including an overview of recent applications in this area from our laboratory.

**(O-8)****MOLYBDENUM IN BEANS: ALLY OR ENEMY OF THE CONSUMER***Katarzyna Wrobel, Kazimierz Wrobel, Alma Rosa Corrales Escobosa**Chemistry Department, University of Guanajuato, 36000 Guanajuato, Mexico**e-mail: katarzyn@ugto.mx*

Molybdenum is an essential nutrient in organisms across the three domains of life. Catalytic activity of all eukaryotic molybdenum enzymes relies on the molybdenum cofactor (Moco), which is synthesized in mitochondria. In Moco, Mo is covalently bound to two sulfur atoms within a tricyclic pterin moiety, referred to as molybdopterin. Dietary exposure to molybdenum has rarely been associated with toxic effects in humans; however its antagonistic interaction with copper has been often reported. In particular, the individuals with inadequate intake of dietary copper or some dysfunction that makes them copper-deficient could be at greater risk of molybdenum toxicity. Formation of stable ternary complexes between thiomolybdate species and both, free and protein bound copper under physiological conditions is well established and thiomolybdates are currently used in chelation therapy of Wilson disease to eliminate the excess of Cu accumulated in liver and brain. On the other hand, it was postulated that molybdenum overload is a main cause of the secondary deficiency of copper in ruminant animals, leading to the oxidative damage and the disturbed carbohydrate metabolism. It has also been demonstrated that molybdenosis can be controlled by maintaining adequate Mo/Cu ratios in the rumen diet. Since disturbance of copper homeostasis in humans has been associated with diabetes, we have pursued possible involvement of Mo as Cu antagonist.

We have studied diabetic patients recruited in central Mexico and we found elevated concentrations of molybdenum and copper in subjects with advanced diabetes. Furthermore, the elimination of copper via urine was directly correlated with biomarkers of typical complications of this disease. In this same study we observed that Cu/Mo ratio in urine enhanced significantly with the progress of nephropathy which points on Mo-aided removal of copper from tissues and its elimination in urine. These results also suggest that Mo-Cu antagonism might be specifically involved in the development of complications in diabetes. In regard to typical local diet, principal component analysis showed a direct association between legume consumption and diabetes incidence/progression. As compared to other dietary sources, these vegetables contain relatively high Mo concentrations (1 - 10  $\mu\text{g/g}$ ), so we first determined total Mo and Cu in different types of beans from local markets and then, the fractionation scheme was implemented in order to evaluate element distribution between soaking water, cooked legumes and broth. In vitro gut model was also applied to assess bioavailable fraction of both elements in cooked beans. Whereas “flor de mayo” and “pinto” beans presented molar ratio of bioavailable Cu/Mo in the range 1.5 - 2.5, in “negro” and “peruano” beans this ratio was significantly higher (5.1 – 8.0). It should be mentioned that Cu/Mo >2 in forage is considered safe for ruminant animals at risk of molybdenosis, therefore low Cu/Mo bioavailable ratio in certain beans might contribute in disturbance of Cu homeostasis, especially in diabetic patients. To gain an insight on possible Cu-Mo interaction in clinical diabetes conditions, human albumin was incubated with glycation agents and binding of Mo and Cu to such modified protein was studied by size exclusion chromatography with ICP-MS detection. It was observed that the elution profiles of both elements were different between non-modified and modified albumin. The results obtained call for more detailed studies on putative role of Mo and Cu in the progression of diabetes.

**(O-9)****DEVELOPMENT OF A DUAL CONCENTRIC INJECTOR (DCI) ICP TORCH COUPLED TO A NOVEL, MICRO, CELL-IN-CELL LASER ABLATION CHAMBER FOR HIGH SPEED, HIGH EFFICIENCY TRANSPORT OF A LASER INDUCED AEROSOLS***David N. Douglas<sup>a,b</sup>, Amy J. Managh<sup>a</sup>, Grant Craig<sup>a</sup>, Helen J. Reid<sup>a</sup> & Barry L. Sharp<sup>a</sup>**<sup>a</sup> Centre for Analytical Science, Department of Chemistry, School of Science, Loughborough University, Loughborough, Leicestershire, LE11 3TU, UK**<sup>b</sup> Inorganic Analysis, NMI Operations, Science and Innovation, LGC Group, Queens Rd, Teddington, Middlesex, TW11 0LY, UK**E-mail: [b.l.sharp@lboro.ac.uk](mailto:b.l.sharp@lboro.ac.uk)*

Chemical imaging, single cell analysis<sup>1</sup> and nano-particulate analysis are applications for which laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) is finding growing use. However, current laser cell designs limit the applicability of this analysis technique to these areas. A significant limitation is the long washout times of commercial cells (the spatial dispersion of the aerosol resulting in a temporally disperse signal of commercial cells). To resolve spatial information long periods between sampling must be employed, or a sacrifice of spatial resolution must be made by monitoring the signal from mixed successive ablation sampling points. Temporal dispersion also results in a poorer sensitivity and thus a reduced signal to noise ratio. For small sample masses this becomes critical; where increasing temporal dispersion for a discreet sample results in the signals approaching the limit of detection.

We present a novel ICP torch and laser ablation cell, the Dual Concentric Injector (DCI)-Sniffer, designed and evaluated at Loughborough University, that minimises aerosol dispersion and results in fast-efficient transport of the laser induced aerosol. A holistic approach was taken to design a single conduit from point of sampling to introduction of the aerosol into the plasma. In brief a cell-in-cell design is employed, using a micro-capture device (the Sniffer); to encompass the ablation aerosol and fix the sampling point relative to the cell outlet, such that response is uniform for sampling. The micro-capture device is coupled to an ICP torch using a short single conduit of fused silica to minimise aerosol dispersion. The transport conduit forms part of the ICP torch, and facilitates injection of the aerosol directly into the plasma, using a single transport gas of helium at a flow rate of *ca.* 0.1 Lmin<sup>-1</sup>. This conduit lies within a secondary injector in which argon (termed the sheath gas) is used to form a stable sample channel. The gas also provides cooling of the transport conduit, such that it can be extended towards the plasma and past recirculation points described previously by modelling<sup>2</sup>.

The design described is shown to produce peaks of <10 ms FWHM when ablating NIST SRM glass 612

*Amy J. Managh, Sheldon L. Edwards, Andrew Bushell, Kathryn J. Wood, Edward K. Geissler, James A. Hutchinson, Robert W. Hutchinson, Helen J. Reid and Barry L. Sharp, *Anal. Chem.*, 2013, **85** (22), 10627-10634**Helmut Lindner, Ayrat Murtazin, Sebastian Groh, Kay Niemax & Annemie Bogaerts, *Anal. Chem.*, 2011, **83** (24), 9620-9266*

**(O-10)****METAL COMPLEXES SPECIATION ASSISTED BY ICP MS: MOVING FRONTIERS**

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In term of inorganic element speciation by mass spectrometry, the hardest chore has always been the study of unstable species to which metal complexes belong. Because the use of sampling and separation techniques is necessary to insure speciation analysis of natural sample by ICP MS, it often results in total or partial loss of original metal complexes.

To circumvent this issue, progresses had to be done in adapting sample preparation, developing soft separation techniques and using cutting edge high resolution molecular MS instrument to assist ICP MS detection. At first, analysed samples should be in the same state as *in vivo* or as close as possible because tissue homogenisation, strong dilution, lyophilisation can alter drastically metal complexes content. Therefore, micro-sampling, fluids or homogeneous samples are privileged. Then, appropriate chromatographic (or electrophoretic) methods have to be optimized for the searched species. Because of their usually good metal complex recovery and complementary mode of separation, hydrophilic interaction chromatography (HILIC), reverse phase chromatography (RPC) and size exclusion chromatography (SEC) turned out to be the most suitable techniques especially when different buffers (pH, salt composition...), columns and separation techniques are compared for a same sample. Finally, the continuous progress of Fourier transform based molecular mass spectrometry offers unparalleled resolution, accuracy of mass measurement, and intrascan dynamic range for the analysis of biomolecules. Once peak numbers, retention times and coeluting metals are known because of ICP MS detection, it gives a clear advantage to detect low concentrated metal complexes in raw samples even after using a single chromatographic dimension.

As discussed in this lecture, a fast comprehensive speciation *de novo* of metal complexes in basically all kind of biological matrices can then be accessible.

**(O-11)****ORGANIC AND INORGANIC SELENIUM SPECIES IN STRONGLY-REDUCING WATERS***Dirk Wallschläger, Kelly LeBlanc, Jacqueline London & Matthew Smith**Trent University, 1600 West Bank Dr, Peterborough, ON K9J 7B8, Canada**email: [dwallsch@trentu.ca](mailto:dwallsch@trentu.ca)*

Selenium (Se) is studied intensively in aquatic ecosystems impacted by industrial Se discharges, due to its propensity to bioaccumulate in aquatic food chains and cause reproductive effects in predatory fish and water fowl. It is well known that the redox conditions of ambient waters strongly affect the speciation, bioavailability and mobility of Se, with reducing conditions typically enhancing Se bioavailability through formation of reduced organic Se species, despite the simultaneous formation of insoluble reduced inorganic Se species. Although it is evident that the measurement of discrete reduced Se species in waters is essential for a proper understanding of Se biogeochemistry in such environments, analytical methods for this purpose are surprisingly underdeveloped. In this presentation, we show some analytical improvements with respect to the determination of reduced organic and inorganic Se species in waters, and present some results obtained in natural waters and hydrochemical model solutions.

Reduced inorganic Se species were synthesized from elemental Se by reaction with a number of different Se or sulfur compounds. Analyses were performed by anion-exchange chromatography coupled to inductively-coupled plasma-mass spectrometry (AEC-ICP-MS), with the entire sample preparation and separation conducted in a glove box. We will show the first chromatographic evidence of the existence of dissolved selenide Se(-II) in strongly reducing waters. However, the developed method is not suitable for quantitative determination of Se(-II), because selenide appears to convert into elemental Se during the separation, and thus yields non-quantitative response. A number of additional anionic Se species were observed in some reaction mixtures, which – based on their chromatographic behavior in comparison to sulfur analogs – appear to be Se analogs of polythionates.

Reduced organic Se species were produced by bacteria in anaerobic bioreactors treating industrial effluents containing inorganic Se oxyanions. We will present AEC-ICP-MS methods capable of quantifying discrete organic Se species in waters at low ng/L concentration levels, as well as molecular mass spectrometry techniques for identifying such Se species at the same low concentration levels. We will show that reducing bioreactors produce the Se amino acid selenomethionine, and that it transforms subsequently in the effluent into a number of oxidation products. A number of other, currently unidentified Se species were also observed in these bioreactor effluents, which – based on their chromatographic behavior – appear to be other amino acid-like Se species.

Based on the presented observations, a revised model of Se biogeochemistry and speciation in reducing waters will be presented.

**(O-12)****ARSENOLIPIDS IN MARINE SAMPLES - STATUS AND ANALYTICAL CHALLENGES**

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Arsenic is an ubiquitous element that is present in the environment due to natural and anthropogenic processes. Marine samples are generally more concentrated in arsenic than terrestrial samples, with concentrations typically in the range of 1 to 100 mg kg<sup>-1</sup>. Arsenic has a complex chemistry and up to 100 naturally occurring arsenic species have so far been identified, both water-soluble and lipid-soluble compounds. Most research on arsenic and its chemical forms has so far focused on the water-soluble species, and a large set of data on occurrence and species exist. During the last decade an increased interest in the lipid-soluble arsenic species; the arsenolipids, has been seen.

The most common techniques within arsenic speciation include use of high performance liquid chromatography coupled to the inductively coupled plasma mass spectrometry (HPLC-ICP-MS). However, for speciation analysis of arsenolipids, where organic solvents are required for the separation of species, the ICP-MS needs to be modified by addition of oxygen and use of low solvent flow. A modified ICP-MS set-up for analysis of intact arsenolipids was first applied in 2005<sup>1</sup>. Since then, around 40 intact arsenolipids have been characterised in oils of fish, fish liver and marine algae. In this presentation, the current status and analytical challenges concerning quantitative and qualitative analysis of arsenolipids in marine oils will be discussed.

<sup>1</sup> *Schmeisser, E., Goessler, W., Kienzl, N., and Francesconi, K. A. (2005). Direct measurement of lipid-soluble arsenic species in biological samples with HPLC-ICPMS. *Analyst*. **130**: 948-955.*



**(O-13)****NEW POSSIBILITIES FOR ULTRA TRACE MULTI ELEMENT AND SPECIATION ANALYSIS IN ENVIRONMENTAL SAMPLES USING ICP-MS-MS***Daniel Pröfrock**Helmholtz-Centre-Geesthacht, Institute of Coastal Research, Max-Planck-Str. 1, 21502 Geesthacht, Germany**e-mail: [Daniel.Proefrock@hzg.de](mailto:Daniel.Proefrock@hzg.de)*

The quantification of pollution levels of different environmental compartments such as sediments or the water phase with either organic or inorganic contaminants as well as the measurement of individual body burdens of selected organisms at different trophic levels represents the “standard procedure” used, to derive information with respect to environmental assessment or for the status description of marine ecosystems.

Even though many contaminants show only low concentrations in the environment they are of great concern due to their specific chemical properties and effects even at low dose, which makes highly sensitive analytical methods mandatory. On the other hand side bio molecules such as selected metal proteins gain some interest as potential biological marker for effects of changing environmental conditions (e.g. due to increased contamination levels). Such applications require in particular accurate independent quantification methods e.g. via utilizing the metal content of such proteins or heteroatoms such as sulphur, which are often present at a fixed, known stoichiometry.

Quantitative analysis of elements and element species in marine environmental samples is in particular challenging due to the complex matrix of such samples, which often results in strong interferences when using ICP-MS as detector. Especially the on-going methodological and instrumental developments in analytical chemistry in particular in the field of ICP-MS, which ends up in the recent introduction of ICP-MS-MS, strongly enhanced the possibilities in terms of accurate analysis of such complex samples.

This contribution will highlight the recent possibilities of using ICP-MS-MS in combination with a discret sample introduction approach for the analysis of challenging environmental matrices such as seawater as well as the potential of ICP-MS-MS technique for speciation analysis, with a special focus on two recent HPLC applications, which benefit from the improved sensitivity of ICP-MS-MS compared to conventional cell based ICP-MS as well as the possibilities of the MS-MS configuration when using reactive cell gases.

Within this context a new approach for the sensitive analysis of Gd containing contrast agents has been developed to overcome the challenges related with the application of HILIC-LC. In addition results will be shown related to the application of HPLC-MS-MS as well as the O<sub>2</sub> mass shift mode for the sulfur specific detection of environmental relevant protein biomarkers such as metallothionein in marine environmental samples.



**(O-14)****APPLICATION OF THE ISOTOPIC DILUTION SPECTROMETRY FOR THE QUANTIFICATION OF EXAVALENT CHROMIUM IN CONTAMINATED SOIL SAMPLES FROM NORTHERN ITALY**

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The aim of the work was to detect and quantify hexavalent Chromium in fourteen soil samples coming from a land in the Regione Lombardia (Northern Italy) contaminated by two water plumes containing this toxic form of the element. To do this, we chose to apply an innovative technique: the Isotopic Dilution Mass Spectrometry, that allows the quantification of Chromium (VI) in soil samples, adding a spike of a solution in which the element is enriched in an isotope of minor abundance.

Following the procedure shown in the publication “*Fast and accurate procedure for the determination of Cr(VI) in solid samples by Isotope Dilution Mass Spectrometry*”, samples are subjected to extraction with a focalized microwave system, to a chromatographic separation, and an ICP-MS quantification.

The application of this procedure of extraction to these soil samples evidenced high reduction of the spike to Cr(III), avoiding the quantification. It is well known, in fact, that some compounds and elements in the soil can provoke the complete reduction of the Cr(VI). These are principally Organic Matter, Fe(II) and sulphides.

In order to verify the real influence of these compounds during the extraction steps, we chose some soil samples coming from the whole Lombardia territory with different characteristics, and we analyzed it. We tried to improve the analytic method changing the extraction way, verifying the presence and the level of reduction of the spike solution, using also the SRM NIST 2701. After the application of different extraction conditions (temperature and duration time), we decided to use only one cycle of microwave and a temperature of 80°C, because this was the only condition that allowed to limit the spike reduction under an acceptable level (max 40%), and to obtain a recovery of 96% for the NIST material.

Then the new extraction conditions were applied to our soil samples coming from the polluted land, and after that we were able to quantify the Cr(VI) content, and to appreciate the influence of the Organic Matter and the Iron content over the spike reduction.

The analysis evidenced that the Cr(VI) content in these samples was similar to the one of the other samples not contaminated collected in the whole Region.

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**(O-15)****APPLICATION OF ISOTOPE RATIO ANALYSIS IN ECOSYSTEM RESEARCH**

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After the mind-blowing discovery in 1913 by Thomson and Aston that the element neon consists of atoms of two different masses, isotopes and mass spectrometry have faced a non-stop success story in science. Besides the description of new isotopes of different elements, increased technological improvements in mass spectrometry have led to the discovery of the fact that the natural abundances of isotopes in nature are not stable but are varying due to radiogenic and nucleogenic reactions or due to mass dependent or mass independent fractionation processes. Parallel to the improvement of instrumental precision, an increasing number of isotopic systems has been described for their significant natural variation.

Quite soon, ecosystem research noticed the usefulness of this information in order to investigate natural processes and ecosystem cycles. This has led to the development and application of tools of analytical chemistry to study dynamic biological and ecological processes within ecosystems and across ecosystem boundaries. Since the 1960s/70s the so-called traditional isotopic systems (C, H, O) have been widely applied in ecosystem research shortly followed by N and S, mainly using isotope ratio (gas source) mass spectrometry. The application of thermal ionization mass spectrometry has soon augmented the number of isotopic systems investigated in ecosystem research. Since the advent of MC ICP-MS in the 1990s, a steady increase in ‘non traditional systems’ under investigation has been observed. Until today, the natural variation of almost 50 different isotope systems has been investigated by ICP-MS, so far, with – besides Sr, Pb and U – Ca, Mg, Cd, Li and Hg being the most prominent. The advent of high-resolution instruments was a significant development. New insights in traditional systems such as S and Si could be gained increasing their use in monitoring elemental cycles in e.g. forestry ecosystems or aquatic ecosystems linking elemental cycles to e.g. erosion or climate change. In order to enhance the knowledge gained by isotope research, isotopic data have been translated into (time resolved) isoscapes allowing for the interpretation of elemental cycles, fluxes or the prediction of future scenarios.

Besides the natural variation of the isotopic composition in the environment, the ‘new’ systems have shown augmented potential by using enriched isotopic spikes for the study of ecosystem processes. This is of major importance as the element as such can be monitored. The high sensitivity and instrumental precision of modern mass spectrometer allow for the introduction of low amounts of spike material without significantly disturbing the natural equilibrium. Therefore, natural cycles can be monitored, the fate of elements can be described or spiked natural compartments (water, soil, plants, animals) can be followed through ecosystems.

The lecture will highlight past and present achievements applying isotopes in ecosystems along with an insight into the remaining analytical challenges to avoid conclusions to be drawn from analytical artefacts.

**(O-16)**  
**ISOTOPE ANALYSIS AT TRACE AND ULTRA-TRACE LEVELS IN ENVIRONMENTAL MATRICES**

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High precision isotope ratio measurements by ICP-SFMS and MC-ICP-MS found increasing use in many branches of science ranging from traditional geochronological applications to elaborative multi-tracer experiments in environment studies. Current limitations for even wider use of isotope tools mainly attributed to analytical challenges in obtaining reliable isotope information for elements present at trace- and ultra-trace levels relevant for uncontaminated environmental matrices or in situations where available sample size is limited. Different aspects of method optimization including boosting instrumental sensitivity, improving analyte utilization by element-specific introduction systems, adopting sample preparation/pre-concentration/separation schemes to accommodate large amount of sample will be discussed and several examples of applications will be presented.

**(O-17)****WHERE DOES IT COME FROM? PB ISOTOPES AND ICPMS IN PROVENANCE ANALYSIS***Tom Andersen and Siri Simonsen**Department of Geosciences, University of Oslo, PO Box 1047 Blindern, N-0316 Oslo, Norway*

Provenance information is important in a range of research fields from geology to archaeology and forensic science. Because of geologically controlled variation in the abundances of the naturally occurring lead isotopes  $^{204}\text{Pb}$ ,  $^{206}\text{Pb}$ ,  $^{207}\text{Pb}$  and  $^{208}\text{Pb}$  in ore deposits, Pb isotope data are widely used as an indicator of the provenance of metals. Analytical data from an object can either be compared to a database of ore deposit data or to models of the Pb isotope evolution of domains within the Earth's crust.

The samples analysed in the present study includes bronze and lead objects from archaeological finds, and silver (i.e. Ag-Cu alloy) coins. Both destructive (total metal dissolution) and non-destructive (acid swab sampling, EDTA extraction) sampling methods have been used. The samples have been analysed by a Nu Plasma HR multicollector ICPMS equipped with a desolvating nebulizer.

Mixing of materials of different origins can take place in nature during formation of an ore deposit, and during extraction and subsequent treatment of metal. Two- and three-component mixing cause data points to spread out along linear or planar arrays in 2D or 3D lead isotope space, where only the endmembers defining the mixing trends carry significant provenance information. Conclusive proof of two-component mixing is given by linear correlations between lead isotope ratios and the inverse of lead concentration. For archaeological objects, Pb concentration data are not generally available, unless total dissolution has been used, or the material is homogeneous enough that electron probe or other non-destructive analytical methods can provide representative concentration data.

Pb in bronze generally comes from the source of Cu. Data from bronze objects from Norwegian Bronze Age localities suggest widespread mixing of metal from sources located in central and southern Europe. Data on lead ornaments from a Viking age Norwegian burial also suggest extensive mixing of metal, which has originated mainly from sources in the British Isles, but with possible contributions from ore deposits in Germany and France.

Silver is a special case, since lead is used as a reagent in its metallurgy. Norwegian 19th century silver coins are known to contain a significant proportion of metal from the Kongsberg native silver deposit, which has a distinct, highly radiogenic lead isotope composition. The lead isotope data from coins show no evidence of a contribution of such lead, but suggest an origin of lead from sources in central Europe (e.g. Harz mountains), with possible minor contributions from Caledonian massive sulphide deposits, which provided copper to the coin alloy.

In order to interpret lead isotope data from metal objects in terms of metal provenance, it is important to keep in mind the isotopic consequences of mixing, and also to understand the metallurgical processes involved in the production of the objects

**(O-18)**  
**ON-LINE ISOTOPE DILUTION FOR ROUTINE MULTI-ELEMENTAL ANALYSIS USING ICP-MS**

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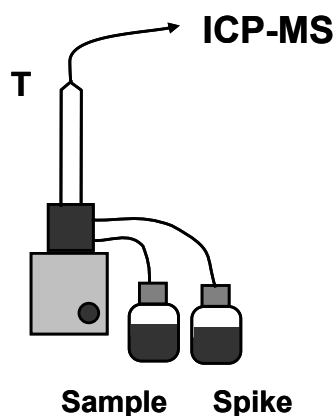
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During the last years, Inductively Coupled Plasma Mass Spectrometry (ICP-MS) has been increasingly used in combination with Isotope Dilution Mass Spectrometry (IDMS) for accurate trace and ultratrace determinations. Isotope dilution is a well-known analytical technique based on the measurement of isotope ratios in samples where their isotopic composition has been altered by the addition of a known amount of an isotopically enriched element. The main analytical application field of IDMS is in the certification of reference materials by National Metrology Institutes. However, there is no scientific or economic reason why IDMS can not be applied also for routine multi-elemental analysis in testing laboratories. Obviously, for the use IDMS in combination with ICP-MS as routine technique for trace element determination several modifications of the “metrological” procedure must be performed.

In this presentation we will describe an alternative procedure for IDMS which can be applied advantageously for routine elemental analysis. First, the classical “batch” mode of operation of IDMS has been changed to an “on-line” mode in which a solution of enriched tracers is mixed continuously with the sample with the help of a peristaltic pump just before the ICP-MS nebulizer. The schematic representation of this mode of operation is shown in Figure 1. In this way, the procedure can be easily automated with the use of an autosampler where standards, samples, blanks and quality controls are located as in any other routine analytical procedure. The main advantages of on-line IDMS are that the calibration graph is no longer required and that the method corrects for matrix effects as well as signal drift, eliminating the need for periodic recalibration and sample re-runs, saving precious ICP-MS time and increasing the productivity of the laboratory. Also, the determination of mono-isotopic elements is implemented in the procedure by pseudo-IDMS. So, in this presentation we will describe the development and validation of the on-line IDMS procedure for multi-elemental analysis in natural waters, seawaters, food samples and biological fluids which could be immediately applied in routine testing laboratories.

Figure 1. Schematic representation of IDMS in the on-line mode of operation.



**(O-19)**  
**INTERNATIONAL CULTURE PROJECTS – MEASUREMENTS ON SKELETONS;  
THE ICP-MS IN ARCHAEOMETRY**

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The work of the CHART group is centered on archaeometry - chemical and physical measurements of archaeological material. Especially the measurement of skeletal material is a corner stone of our ICP-MS program. The aim of our research on human bones is to investigate provenance, health, and population characteristics in the Medieval European cities. Furthermore diagenetic processes are also registered on the measured material. The measurements include the determination of <sup>24</sup>Mg, <sup>27</sup>Al, <sup>44</sup>Ca, <sup>55</sup>Mn, <sup>56</sup>Fe, <sup>63</sup>Cu, <sup>66</sup>Zn, <sup>88</sup>Sr, <sup>137</sup>Ba and (<sup>206</sup>Pb, <sup>207</sup>Pb, and <sup>208</sup>Pb), where Fe, Cu, and Zn is analysed using a CRI reaction system. Calcium, Ba, and Sr reflect diet and provenance, Mg and Zn are elements under strong metabolic control, and Al, Mn and Fe indicate the presence of various diagenetic processes.

The distribution of the elements in different skeletal tissues has been investigated for two different individuals, giving the sampling strategy for larger population groups, and founding the concept of “chemical life history” – a concept developed by us. The distribution of the elements is equal in similar tissues – like e.g. the compact tissues of the upper arm and the thigh bone - which makes it possible to compare results of different individuals, even though different bones have been preserved. In contrast, the distribution of elements in some other tissues in the body does indeed differ. This fact is utilized in the concept of chemical life history, as different tissues developed during a well-defined period of the individual’s life reflecting the provenance and possible treatment of diseases of the individual at that time.

Future research is aimed at prolonging the timeline of chemical life history, by measuring soil samples taken close to the bones. These samples will represent decayed soft tissues such as liver, lung and kidney, and our measurements of mercury on such soil samples show promising results, giving indications of further knowledge to be gained of past populations.

**(O-20)****PNEUMATIC NEBULIZERS: A VERSATILE ANALYTICAL TOOL FOR INTEGRATING ATOMIC AND MOLECULAR MASS SPECTROMETRY FOR THE ANALYSIS OF ELEMENTAL SPECIES, METALLOPROTEINS AND ENGINEERED NANOPARTICLES***Spiros A. Pergantis**Department of Chemistry, University of Crete, Voutes Campus, 71003 Heraklion, Greece**e-mail: [spergantis@chemistry.uoc.gr](mailto:spergantis@chemistry.uoc.gr)*

Pneumatic nebulizers have gained a prominent role in ICP-MS, assisting it into becoming a vital analytical tool in numerous applications which span a wide range of research activities. More specifically, various types of pneumatic nebulizers have already been used to efficiently couple HPLC to ICP-MS, ranging from conventional mL/min mobile phase flows down to nL/min flows. Recent progress for improved coupling of capillary electrophoresis with ICP-MS via pneumatic nebulization has also been demonstrated. Such developments have extended the application of ICP-MS from small molecule elemental speciation analysis to metalloproteomics and proteomics applications. Also, pneumatic nebulizers have recently been demonstrated to provide a suitable means for introducing nanoparticle-containing solutions into ICP-MS for single particle analysis. These areas of research have all been well investigated by the atomic spectrometry community, which continues to invest heavily in their further development.

What has been largely ignored, however, by the broader analytical community is that the pneumatic nebulizer, combined with a mass spectrometer, can also produce bare, gas phase analyte ions. The technique that has been developed using just the pneumatic nebulizer for ion formation is named sonic-spray ionization MS and can be used to analyze a wide range of analytes (practically all the compounds that electrospray can analyze).<sup>1,2</sup> In the case of metal species SSI-MS even provides some interesting advantages over electrospray as it does not use any electrical potentials and thus redox active species can't be affected in any way.

In this presentation we will be discussing both our advances in developing, characterizing and using modified pneumatic nebulizers for the nanoLC ICP-MS analysis of metalloprotein and nanoparticle determination, as well as how such nebulizers perform for molecular MS. Finally, our ambition and efforts to produce an integrated atomic and molecular analysis approach via the pneumatic nebulizer will be discussed.

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**(O-21)****ORGANIC SAMPLES ANALYSIS BY ICP-MS: WHICH INTRODUCTION DEVICE?***Amélie Leclercq<sup>1</sup>, Anthony Nonell<sup>1</sup>, Carole Bresson<sup>1</sup>, Laurent Vio<sup>1</sup>, Frédéric Chartier<sup>2</sup>*<sup>1</sup> CEA, DEN, DANS, DPC, SEARS, LANIE<sup>2</sup> CEA, DEN, DANS, DPC

Nowadays, inductively coupled plasma mass spectrometry (ICP-MS) became a common analytical technique for trace element analysis. However, its use is mostly focused on aqueous samples while organic samples analysis remains scarcer. The increasing development of separation techniques, like liquid chromatography, hyphenated with ICP-MS, as well as the remaining need of organic samples analysis in many fields (non-water compatible samples, in the petroleum industry for instance) stress the importance of a better understanding of the introduction of organic solvents into ICP and promoting this topic.

The introduction of high level of organic solvents is challenging and results in the degradation of the analytical performances of the ICP. For example, high carbon levels can result either in plasma instabilities, sensitivity losses, spectral and non-spectral interferences, contaminations and cone orifices clogging. Also, organic vapors can lead to the plasma switching off. Various introduction devices are commercialized to address the issue of aqueous or organic solvents introduction in ICP with the aim to improve the analytical performances, while facilitating the experiments whatever the sample (fast wash out, robust conditions...).

In the field of nuclear waste management, speciation studies of actinides and lanthanides are carried out by chromatography coupled with ICP-MS, involving the use of organic solvents. Additional technical constraints are induced by nuclearized spectrometers, mandatory to handle radioactive samples.

The goal of this study is to compare various introduction devices to bring high level of organic samples in ICP-MS, whilst remaining easy to use. Three introduction devices with different designs and suitable for the nuclear applications were compared: a cyclonic spray chamber (considered as the basic configuration), a Peltier-cooled cyclonic spray chamber with O<sub>2</sub> introduction port (PC<sup>3</sup>, Elemental Scientific) and a desolvation device (Apex from Elemental Scientific).

The study is focused on acetonitrile, as this organic solvent is widely used in chromatography and known to be hardly compatible with ICP sources. The introduction devices are compared regarding robust analytical performances and according to the maximum tolerable uptake rate of high level of organic samples in the ICP. The sensitivity and stability measured for various analytes have been compared for the three introduction systems. Finally, the cost and ease-of-use, especially for nuclear applications, of these devices will also be discussed.



**(O-22)****ICP-MS: ADVANCEMENTS DUE TO EVER-INCREASING CHALLENGES***Meike Hamester<sup>1</sup>, Rene Chemnitzer<sup>1</sup>, Iouri Kalinitchenko<sup>2</sup> and Soren Dalby<sup>3</sup>*<sup>1</sup>*Bruker Daltonics, Fahrenheitstrasse 4, 28359 Bremen, Germany*<sup>2</sup>*Iouri Kalinitchenko, 3500 W Warren, Fremont, CA 94538, USA*<sup>3</sup>*Bruker Daltonics, 39 Kallerupvej, 2640 Hedehusene, Denmark**e-mail: [meike.hamester@bdal.de](mailto:meike.hamester@bdal.de)*

ICP-MS is one of the fastest growing analytical techniques passing other atomic spectroscopy techniques such as atomic absorption and ICP optical emission spectroscopy. Environmental applications are still representing the largest application field, but followed by geochemistry, material science, clinical analysis and of course semiconductor related applications. Especially in recent years the ICP-MS market has and is still experiencing a boost due to new fields of applications: analyses of nanoparticles, laser ablation for imaging applications, increasing demand for speciation analyses which requires seamless HPLC or coupling with Gas Chromatography devices. A lively exchange between researches, operators and instrument companies is essential to ensure requirements are met and innovation is driven.

New and broader fields of applications – e.g. in pharmaceutical analysis - often require analyzing more complex sample types, speed of analysis in combination with sensitivity is key for nanoparticle determination as well as for laser ablation investigations in geochemistry and clinical research. Superior abundance sensitivity a must for isotope ratio analysis. Beside those characteristics a robust plasma which decomposes the sample, delivers a narrow kinetic energy distribution and low oxides, is mandatory as well.

The presentation will describe the unique quadrupole based ICP-MS (Bruker). Key elements are a 90° 3D reflecting ion lens, which will focus ions of different kinetic energies with highest efficiency to the mass analyzer, robust plasma generation, which reflects the needs of a modern and versatile laboratory, and not to forget an unparalleled strategy to overcome spectral interferences without disadvantages. The presentation will demonstrate the figures of merit of this instrument and furthermore the presentation will show different application examples which benefit from high sensitivity, fast scanspeed and robust plasma generation.

**(O-23)****NEXION FAST FIAS UNLIMITED TDS: DIRECT INJECTION OF HIGH (30%) DISSOLVED SOLID SAMPLES***Olve Frederichsen<sup>1</sup> and Paul Watson<sup>2</sup>,**<sup>1</sup> PerkinElmer As Norge, Oslo, Norway, <sup>2</sup> Elemental Scientific Inc., Omaha, Nebraska, USA.*

The NexION *FAST* FIAS is a novel flow injection system that is capable of handling an unlimited range of Total Dissolved Solids (TDS). The system first loads sample into a loop, then rapidly switches the valve to inject micro volumes of sample into the plasma. The resultant fast sample injections have short residence times (3 sec) in the plasma and greatly reduce cone deposition for even the highest TDS (30% brine) samples. Minimal matrix suppression for 125ms injections permits undiluted solutions containing up to 30%TDS (brine) to pass EPA internal standard recovery limit. Precise (< 2%) data for transient signals generated from 4  $\mu$ L sample injections (and smaller) is obtained by integrating peak area. Accuracy for 125 ms injections is illustrated through the generation of linear calibration curves (>0.9999). Most significantly, signal response for rapid injections is constant regardless of TDS, suggesting near elimination of matrix suppression effects. Therefore, a simple calibration curve (prepared in 3% HNO<sub>3</sub>) can be used to determine elemental concentrations for a full range of TDS solutions. The accurate recovery of Pb spiked into a 30% brine solution based on a calibration curve in 3% HNO<sub>3</sub> indicates *FAST* FIAS eliminates the need to dilute or matrix match high TDS solutions.

**(O-24)****NEW APPROACHES AND APPLICATIONS IN GLOW DISCHARGE SPECTROSCOPY***Steven J. Ray, Andrew Schwartz, Elise Dennis, Andrew Storey, and Gary M. Hieftje.**Department of Chemistry, Indiana University, Bloomington, Indiana, USA.**e-mail: [sjray@indiana.edu](mailto:sjray@indiana.edu)*

This presentation will explore several new adaptations of the multitalented glow discharge (GD) for analytical spectroscopy. In one example, further development of a novel liquid-sampling GD known as a solution-cathode glow discharge (SCGD) will be examined. The SCGD is an atmospheric-pressure glow discharge that is sustained in the open atmosphere between an anode pin and a sample solution, which acts as the discharge cathode. The plasma lies directly atop the surface of the analyte solution, and liquid sampling occurs by a mechanism akin to sputtering that ejects sample material into the SCGD plasma. There, analyte species are desolvated, atomized, and excited, and the constituent atoms observed by atomic emission spectroscopy (AES). The SCGD-AES technique offers a number of significant advantages over conventional plasma emission techniques. Introduction of the analyte solution into the plasma occurs by a direct introduction mechanism, which promises high efficiency and rapid response. Further, because the surface of the flowing solution cathode is self-renewing, memory effects are minimized. The SCGD also operates in open atmosphere, employs a very simple experimental setup, requires no compressed gases, and uses only 70W of power. Thus, it well suited for portable instrumentation, and its simple construction and glow discharge structure make it amenable to miniaturization. The experimental construction of the SCGD, its operating mechanisms, spectroscopic characteristics, and analytical figures of merit of this SCGD-AES instrument will be presented, and the use of the SCGD in several applications examined.

A second discussion will explore a new approach to mass spectrometry known as the Distance-of-Flight Mass Spectrometer (DOFMS) and its application in atomic spectrometry. In many ways, DOFMS is the complementary technique to time-of-flight mass spectrometry (TOFMS). In TOFMS, ions given the same energy achieve a  $m/z$ -dependent velocity, and ion  $m/z$  is measured based upon the time required for to traverse a set distance. Complementarily, DOFMS measures the distance each  $m/z$  is able to travel during a specific time by employing a position-sensitive ion detector. Like TOFMS, the DOFMS is architecturally simple, rapid, and has an unlimited  $m/z$  range. However, DOFMS are able to employ new solid-state array ion detectors to great advantage, providing greater detection efficiency and dynamic range than typical TOFMS, and obviating the need for fast electronics and timing circuits. Interestingly, both DOFMS and TOFMS can be fused into a single instrument to provide greater analysis flexibility and new capabilities. As important, the DOFMS strategy permits new experiments to be accomplished not possible with typical TOFMS. For example, a new technique known as Zoom-TOFMS is made possible by using the ion focusing theory of DOFMS within the TOFMS framework. In Zoom-TOFMS, a small window of the mass spectrum can be analyzed with increased mass resolution to overcome isobaric overlaps without loss in S/N. The theory of operation and experimental advantages of DOFMS techniques will be discussed, and the analytical performance of this new type of mass spectrometer will be described.

**(O-25)****DEPTH PROFILE ANALYSES BY GLOW DISCHARGE MASS SPECTROMETRY AFTER ION IMPLANTATION***Marisa Di Sabatino, Chiara Modanese, Lars Arnberg**Dept Materials Science and Engineering, Norwegian University of Science and Technology (NTNU), A. Getz vei 2B, 7491 Trondheim, Norway.**Email: [Marisa.di.sabatino@material.ntnu.no](mailto:Marisa.di.sabatino@material.ntnu.no)*

Glow discharge mass spectrometry (GD-MS) is a well-known technique for bulk concentration analysis, while no generally accepted method of quantification of depth profiles has emerged for this technique. Currently, the GD-MS instruments are receiving increasing attention for the analyses of materials for photovoltaic (PV) applications. Accurate measurements of doping elements, such as B and P, as well as metallic impurities, such as Fe, Cu and Ti, play a key role for predicting the electrical properties of the device.

In this work we have developed a method to assess the capability of depth profile analysis by GD-MS for several impurities relevant for solar cell silicon. A fast-flow direct-current high resolution glow discharge mass spectrometer has been used. Six multicrystalline p-type silicon samples with contamination of B, P, Fe, Cu and Ti have been investigated. Ion implantation has been used for impurity contamination with a target depth of 3  $\mu\text{m}$ . The acquisition time was approximately 30 seconds, giving a depth resolution of approximately 0.5  $\mu\text{m}$ .

The GD-MS concentration profiles of the samples contaminated with B and P agreed well with the levels implanted, while for Fe, Cu and Ti the concentration profiles did not show clear trends. This indicates that for transition metals, and especially for the fast diffuser Cu, different impurity distribution mechanisms are occurring after ion implantation (e.g. solid-state diffusion, microsegregation, etc).

**(O-26)****NOISE CHARACTERISTICS OF AN ATMOSPHERIC PRESSURE GLOW DISCHARGE WITH A SOLUTION CATHODE UNDER INERT GAS AND AIR ENVIRONMENTS**

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Atmospheric pressure glow discharges with solution cathodes, also known as electrolyte cathode atmospheric glow discharges (ELCAD) or solution cathode glow discharges (SCGD), are novel spectrochemical emission sources that are useful for direct elemental analysis of aqueous samples. Unlike traditional atomic spectrochemical techniques, such as inductively coupled plasma atomic emission spectroscopy (ICP-AES) and atomic absorption spectroscopy (AAS), the SCGD does not require a nebulizer and spray chamber and therefore is not analyte flicker noise limited by the sample introduction system. The simplicity and long term stability of the dc-powered SCGD may allow the technique to be used for real-time monitoring in industrial process control and environmental monitoring scenarios.

Initial stability studies in our lab indicate that the SCGD has excellent long term stability for the alkali metals. However, the noise characteristics of the SCGD will dictate how this technique is implemented. Noise power spectra will be presented for the SCGD operating under a range of power and sample solution flow rates. Correlation between optical noise and power supply fluctuations will be shown. A hollow anode is used to deliver gases (Ar, He, air) to the plasma and noise power spectra will be presented for different plasma gases and flow rates. The characterization and identification of noise types will allow intelligent choices of integration periods and a means of quantifying future system improvements.

**(O-27)****COMPARISON OF FAST-FLOW GD-MS AND GD-OES METHODS FOR DEPTH PROFILING OF CR CONVERSION COATINGS ON FE SUBSTRATE***Karol Putyera<sup>1</sup> and Arne Bengtson<sup>2</sup>*<sup>1</sup>*Evans Analytical Group, Liverpool, New York 13088, USA*<sup>2</sup>*Swerea KIMAB, Isafjordsgatan 28A, SE-164 07 Kista, Sweden**e-mail: [kputyera@eag.com](mailto:kputyera@eag.com)*

Chromium conversion / diffusion coatings are made by depositing a thin layer of chromium coating on a substrate, then subject the material to elevated temperature treatment process in which an alloy is formed by the inward diffusion of chromium into the base metal. The main purposes of the coatings are to achieve specific material properties e.g. corrosion protection, improved paint adhesion or scratch resistance, among others. For instance, chromide diffusion coatings are still widely applied to hot section industrial gas turbine blades and vanes to protect them against high temperature oxidation and hot corrosion. Or, high carbon steels can form a hard dense diffused chromium carbide surface layer which has high hardness and excellent wear resistance.

The quality of a conversion coating is closely linked to the chemistry of the coating and the interface between the coating and the substrate, i.e. the variations of the elemental composition with depth. Therefore, elemental depth profiling techniques are very important tools for characterization of these types of coatings, both for quality control and for process / material development point of view. In this work, fast glow discharge methods for depth profiling of a chromium conversion coating on steel have been developed and compared. The chromium coating is approximately 20 µm, after the conversion, the chemistry of the alloy changes down to about 100 µm. Methods have been developed for the fast flow GD-MS instruments (Thermo Element – GD) and a GD-OES instrument (Leco GDS 850). In general, GD-OES has the speed advantage related to higher sputtering and data acquisition rates and relatively easy handling. Fast flow GD-MS on the other hand has superior sensitivity and simplified standardization requirements. The analytical results using both instruments will be presented and discussed.

**(O-28)****REQUIREMENTS AND CAPABILITIES OF LA-ICP-MS FOR TRACE ELEMENTS ANALYSIS AND IMAGING***Olga Borovinskaya, Sabrina Gschwind, Joachim Koch, Bodo Hattendorf, Hao Wang, Detlef Günther**ETH Zürich, Laboratory of Inorganic Chemistry, Vladimir-Prelog-Weg 1-5/10, 8093*

Laser ablation as a direct solid sampling technique in combination with ICP mass spectrometry has proved to be successfully applicable for major, minor and trace element analysis and isotope ratio determinations in a variety of samples and the number of instruments installed per year is continuously growing.

An in-house built ICPTOFMS at 33 $\mu$ s temporal resolution was used to study single pulse ablations and the signal were quantified using liquid calibration introduced via micro droplet generator. The high temporal resolution allows studying downhole fractionation in great detail. First results on multiple laser pulses will be presented. Furthermore, the signal manipulation for high spatially resolved imaging will be discussed.

Increased flexibility in laser ablation has always been limited due to the requirement of an airtight aerosol transport system to the ICP. A recently developed gas exchange device (GED) circumvents these requirements and makes sampling in air possible. The use of such a Q-GED system in combination with a portable laser ablation system, allowing the ablation of samples of any size and geometry, was tested for archeological samples and will be presented.

Finally, more research on the flame synthesis-based production and characterization of homogeneous calibration materials for LA-ICPMS was carried out and some data for carbonates will be presented.

**(O-29)****IMPROVED RESOLUTION AND PRECISION FOR ELEMENTAL IMAGING OF GEOLOGICAL SAMPLES USING LA-ICP-MS***Rene Chemnitzer<sup>1</sup>, Søren Dalby<sup>2</sup>, Meike Hamester<sup>1</sup>**<sup>1</sup>Bruker Daltonics, Bremen, Germany; <sup>2</sup>Bruker Daltonics Scandinavia, Solna, Sweden*

The combination of sample preparation techniques, separation techniques and sample introduction techniques with ICP-MS provides multidimensional information about various sample types.

Hyphenated techniques are more often part of the routine work in laboratories and there is an increasing need for lower detection limits and better resolution. The application of laser ablation for elemental mapping requires a fast transport and wash-out of the laser generated aerosol from the ablation cell and at the same time also a complete acquisition of all isotopes of interest in the ICP-MS. Since the material available for the analysis is limited and there is a clear tendency towards smallest possible spot sizes – the combination of high sensitive and fast data acquisition is the key for better data quality. New instrumental developments allow resolutions to single-digit  $\mu\text{m}$  spots and new ablation cells show improved transport characteristics. The presentation describes the layout of an ICP-MS instrument that offers unmatched high sensitive and fast scan speed for this kind of application. Preliminary results on reference materials NIST 612 show more than 2Mcps for Th and U ( $\varnothing 65\mu\text{m}$ , 10J/cm<sup>2</sup>, 20Hz) at very low oxide formation of 0.3% ThO/Th. Limits of detection are calculated to 0.04 and 0.08 ppb for Th and U. The laser ablation parameters were optimized for the ablation of geological. The ICP-MS instrument was optimized to high sensitivity to allow the ablation with small spot sizes.

The work presents results from a topological mapping experiment on an ammonite sample that show a very detailed distribution of Ca and Sr but also trace elements like Sc, Y and Zr. Parameter including spot size, sample introduction, 3D-ion focusing and scan speed are discussed.



**(O-30)****SAMPLE PREPARATION STRATEGIES FOR FOOD AND BIOLOGICAL SAMPLES PRIOR TO NANOPARTICLE DETECTION AND IMAGING***Erik H. Larsen and Katrin Loeschner,**Technical University of Denmark, National Food Institute, 19, Mørkhøj Bygade, Bldg. B, DK-2860 Søborg, Denmark; [ehlar@food.dtu.dk](mailto:ehlar@food.dtu.dk)*

Accurate and precise characterization of metrics such as size, mass, shape etc. of nanoparticles (NPs) remains a challenging task. In order to determine quantitative metrics that are relevant in food monitoring or in risk assessment, an instrumental separation method like asymmetric field flow fractionation (AFFF, or AF<sup>4</sup>) coupled on-line to various detectors including static and dynamic light scattering (LS), UV or fluorescence (FL) spectroscopies and ICP-MS have proven useful and powerful [1, 2, 3]. Furthermore, additional information obtained by an imaging method such as transmission electron microscopy (TEM) proved to be necessary for trouble shooting of results obtained from AFFF-LS-ICP-MS.

Aqueous and enzymatic extraction strategies were tested for thorough sample preparation aiming at degrading the sample matrix and to liberate the AgNPs from chicken meat into liquid suspension. The resulting AFFF-ICP-MS fractograms, which corresponded to the enzymatic digests, showed a major nano-peak (about 80 % recovery of AgNPs spiked to the meat) plus new smaller peaks that eluted close to the void volume of the fractograms. Small, but significant shifts in retention time of AFFF peaks were observed for the meat sample extracts and the corresponding neat AgNP suspension, and rendered sizing by way of calibration with AgNPs as sizing standards inaccurate.

In order to gain further insight into the sizes of the separated AgNPs, or their possible dissolved state, fractions of the AFFF eluate were collected and subjected to ICP-MS analysis in single particle (sp) mode. The results showed that the first eluting AFFF peaks contained some dissolved Ag-species and the later eluting peaks primarily contained AgNPs of increasing sizes.

Finally, the possibility of using alkaline pre-treatment of rat spleens prior to sp-ICP-MS analysis of their content of gold nanoparticles (AuNPs) was tested and compared with enzymatic sample preparation [3]. The results showed that the same results, with respect to the obtained number-based size distribution for AuNPs, were obtained for the two preparation methods. In contrast, the alkaline method was by far superior for quantification of AuNPs and was comparable with that obtained by ICP-MS after digestion of the samples in *aqua regia*. The reason for this is however, not fully understood, and requires further study.

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**(O-31)****PUSHING THE LIMITS OF SINGLE PARTICLE ICP-MS FOR DISCRIMINATING AND SIZING SMALL NANOPARTICLES IN ENVIRONMENTAL SAMPLES***Martin Hassellöv, Jani Tuoriniemi and Geert Cornelis**Department of Chemistry and Molecular Biology, University of Gothenburg, SE-41296 Gothenburg, Sweden**e-mail: martin.hasselov@chem.gu.se*

Due to the often special reactivities of nanomaterials, there are rapidly increasing development of them in a range of technology and market sectors. This massive research effort is putting high demands on method development for detection, quantitative analysis and physico-chemical characterization, both in terms of applications but for their potential human or ecosystem risks. It is especially nanoparticles that are giving rise to risk concerns, due to their small size and special properties (1). Although many physicochemical properties can be important for the understanding the material properties, the most fundamental would be size and concentration. To understand particle dynamics and also toxicology it is believed that number based measurements provide the most relevant measurement. The European Commission has also made a regulatory recommendation that classification of nanomaterial should be founded on number based size distributions.

Although there are many techniques available that can fulfill this task in powders or simple solutions, there are very few that can be applied to real world samples. Single particle ICP-MS (spICPMS) is one of the few that are capable of detecting, enumerating and sizing metal containing nanoparticles with elemental selectivity at ultra trace levels in complex solutions. This presentation will go through the basic theory of spICPMS, and then focus on the nanoparticle discrimination (2). Generally there is a need to be able to measure as small nanoparticles as possible but in practice the size detection limit for many metal based nanoparticles are limited by number of isotopes, stoichiometry and ionization and ion transmission, but also by the background level of dissolved ions and very small nanoparticles. The lowest detectable size for a given particle with an optimum method will thus ultimately be limited the discrimination of the particle signal from the background. In order to improve the discrimination capability a new approach have been developed that is based on fitting the noise and background signal to a mixed Polygaussian probability mass function and then deconvoluting it from the total signal (3). This signal deconvolution procedure allowed accurate determinations of size distributions of as small as 10 nm Au particles compared to 30 nm particles without. Further will be presented a new fast, true realtime data acquisition mode of spICPMS that also improve the smallest size discrimination and dynamic range of nanoparticle concentration significantly (4).

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**(O-32)**

**HANDLING THE HIGH MATRIX SAMPLES WITH EASE USING NEW ICP-MS.**

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ICP-MS is well accepted techniques for elemental analysis. The new ICP-MS system offers many benefits over traditional elemental spectroscopy techniques extending the analytical capabilities in elemental analysis and offers solutions for many challenges.

Ultra-high-matrix introduction technology with ICP-MS enables laboratories to measure samples containing up to 25 percent total dissolved solids, 100 times higher than the current equipment. This enables direct measurement of sample types previously inaccessible by ICP-MS.

The new ICP-MS has been engineered and optimized to provide elemental laboratories with the industry's most powerful and easy-to-use quadrupole ICP-MS solution. Examples on following features will be described with 2 applications handling high content of TDS.

Unprecedented matrix tolerance - Ultra-high-matrix introduction technology enables laboratories to measure samples containing up to 25 percent total dissolved solids, 10 times higher than the current benchmark held by the 7700 ICP-MS limit. This enables direct measurement of sample types previously inaccessible by ICP-MS.

Enhanced trace-level detection - A novel interface design, optimized expansion-stage vacuum system and new orthogonal detector system (ODS) reduce background and improve sensitivity, dramatically improving signal to noise for 10-fold lower detection limits than any quadrupole ICP-MS system available today.

Widest dynamic range of any quadrupole ICP-MS - Patented ODS technology delivers up to 11 orders of magnitude dynamic range, from sub-ppt to percent-level concentrations, which enables users to measure trace elements and majors in the same run. An industry-first capability, this eliminates analyte-specific tuning and simplifies method development, virtually eliminating over-range results.

**(O-33)****PLASMA BASED TECHNIQUES FOR BIOLOGICAL AND CLINICAL APPLICATIONS***Ewa Bulska**Faculty of Chemistry, Biological and Chemical Research Center, University of Warsaw, Żwirki i Wigury 101, 02-089 Warszawa, Poland**Email: [ebulska@chem.uw.edu.pl](mailto:ebulska@chem.uw.edu.pl)*

The usefulness of the analytical techniques for the investigation of biological and clinical objects grown significantly over the last years. This is due mainly to the improved analytical performance and the ability to provide novel set of the information about the composition of the samples. Several techniques, those involved plasma as an ionization / excitation source are considered to be very useful for the investigation of the elemental as well as isotopic composition of e.g. plants, animal tissues, bacterial strains, urine, blood or saliva.

In the last years a lot of attention was focused on the investigation of the process ongoing in plants and animal tissues towards understanding the specific mechanism, when they are exposed to the various environmental conditions, e.g. toxic substances or drugs. Several analytical scenarios, those involving plasma based instrumental techniques were tested towards investigation of the elements transport as well as its bio-transformation via examination of the presence of specific compounds in various tissues.

Several examples will be given to illustrate growing application areas. Commonly used determination of the elemental composition of tissues will be exemplified by the evaluation of the transport efficiency of selected elements in plants, all towards understanding its response to the environmental conditions. Chromatographic techniques coupled to ICPMS were used to identify chemical forms of selected elements. The analytical scenario for speciation studies which was developed toward the evaluation of the bio-mechanism of plants to adapt for unfavorable environmental conditions will be exemplified by the investigation of the selenium and zinc.

The laser ablation coupled to ICP MS facilitates to perform the elemental distribution over the solids surface and sub-surface domain. This was used for the investigation either the cross section of various tissues (e.g. brain, ovary, uterus) or for the examination of the capillary gel after species separation in blood or serum. This approach will be exemplified by several projects related to the Wilson disease as well as investigation of the effect of platinum based anti cancer drugs.

**(O-34)****ICP BASED METHODS FOR THE ANALYSIS OF ENERGY STORAGE MATERIALS***Sascha Nowak\*, Björn Hoffmann, Britta Vortmann, Jennifer Menzel, Timo Schwieters, Martin Winter**University of Münster, MEET Battery Research Center, Corrensstr. 46, 48149 Germany**\*([sascha.nowak@uni-muenster.de](mailto:sascha.nowak@uni-muenster.de))*

In recent years, lithium-ion batteries (LIBs), first commercialized by Sony in 1991, became the dominating electrochemical power source. An important application of LIBs is energy supply for mobile devices and electrical vehicles. The economic success of these high energy storage systems is explained by the high specific energy and excellent cycling stability. In general, LIBs consist of a lithium metal oxide cathode, a carbonaceous anode, a polyolefin-based separator and the electrolyte, which is mainly composed of a mixture of organic carbonates and a conducting salt. However, when packed in large batteries, such as those required for EV and HEV applications, LIBs are prone to accelerated aging and low safeness. Furthermore, the exact mechanisms are still unknown or, respectively, not verified.

In this work we present the use von inductively coupled plasma based methods for the analysis of energy storage materials. These methods include the analysis of the electrodes and electrolyte systems in terms of elemental analysis or the surface investigation with by means of LA-ICP-MS. Furthermore, the hyphenation to an ion chromatography system for elemental and quantitative information of the electrolyte system will be presented.

**(O-35)****DETERMINATION OF HEAVY METALS IN THE FOOD CHAIN USING ICP-OES SPECTROMETRY***Uwe Oppermann<sup>1</sup>, Jan Knoop<sup>1</sup>, Remko Roolvink<sup>1</sup> and Albert van Oyen<sup>2</sup>**<sup>1</sup>Shimadzu Europa GmbH, Albert-Hahn-Str. 6-10, D-47269 Duisburg, Germany**<sup>2</sup>Carat GmbH, Harderhook 20-22, D-46395 Bocholt, Germany**e-mail: [uo@shimadzu.eu](mailto:uo@shimadzu.eu)*

Food is always a hot topic discussion and in the focus of “state of the art” analytical techniques. Food scandals all around the world from eggs to horsemeat, tainted wine, oil, and milk forces the European community to establish an integrated approach on food control. The target is a high level of food safety, animal health, animal welfare and plant health within the European Union through so called “farm-to-table” measures and monitoring, ensuring the effective functioning of the European market.

Due to the globalization the food is no longer a local product, but transported over thousands of kilometers, from where it is produced to where it is eaten. That’s why food needs preservation to be robust against long transport, needs cooling and sophisticated packaging. Unfortunately the additives for preservation as well as the packaging material are sources for contamination.

Strict and steady control from the origin of the food to the final product is needed to protect consumers against undesired contaminations and guarantee a high level of quality. This is achieved by controlling limits of maximum allowable concentrations of hazardous substances. Recent examples are the European drinking water regulation, the European food safety regulations and the food and packaging directive. In all of those applications it is essential to provide the right “tool sets” in order to support the accurate monitoring of harmful substances (such as mercury, cadmium and lead) in our environment.

These tool sets consist of one or more analytical systems. A wealth of spectroscopic methods exists for residue analysis and quality control. The methods and type of instrumentation employed are dictated by the composition and formulation of the samples to be measured. The most suitable technique for quantitative analysis of heavy metals in food and food packaging is ICP-OES spectrometry. Experimental data of antimony in food and food packaging will be presented and analytical results will be discussed.

**(O-36)****IMPROVED METHODOLOGY FOR CALIBRATING LINEAR RESPONSES IN ROUTINE ANALYSIS***Arngrímur Thorlacius**Agricultural University of Iceland, Hvanneyri, 311 Borgarnes, Iceland**arngrimur@lbhi.is*

A vast majority of calibration work in analytical chemistry involves responses that increase linearly with concentration. For the calibration of linear responses the method of linear least squares regression is used almost exclusively. IUPAC has published guidelines which clearly specify the limiting conditions which this algorithm is based on and the importance of taking these into account, when using it.

One condition is that there should be no errors associated with the concentration or x-variable. This will never be strictly met, but for most practical purposes this is probably not a major concern. A second condition demands homoscedasticity i.e. that the error in the estimated response is constant over the calibrated concentration range. Should your calibration fulfil both conditions you can apply a so-called normal or ordinary regression algorithm and this is what most analysts do. The IUPAC guidelines however firmly stress that before this most simple least squares algorithm can be used one must clarify, preferably through statistical testing, that the above stated conditions are indeed met. If they are not, weighted regression is recommended. Ordinary regression is often praised for its inherent capability to produce error estimates. However, since these estimates are calculated under the assumption of homoscedastic errors they are of little or no value when this condition is not met.

For instrumental analysis, homoscedasticity is rather an exception than a rule and, since normal regression is very widely used, it may be feared that this problem is often ignored. It is the belief of the present author that the conventional use of linear regression, although very widely adopted, should be avoided in routine analytical work. This holds especially for low level determinations. An alternative approach, which has been very thoroughly tested, will be suggested and explained in this lecture. This is essentially a one-point calibration method, measuring one calibration standard and a blank at regular intervals throughout a measurement run. Linear regression is used to estimate both responses (standard and blank), yielding real-time drift-corrected standardisation for each and every sample in the run. The deviation of individual blank measurements from the corresponding regression value provides a basis for the calculation of limits of detection and quantification, respectively. Taking additionally into account the spread of individual standards measurements around the regression line for all standards, one can get a useful estimate of the uncertainty of individual readings. The suggested approach will be demonstrated and explained, using data sets from ICP-OES analysis measuring several elements in a large number of samples.

Compared with conventional calibration the suggested approach yields improved accuracy as well as better estimates for both detection limits and measurement uncertainties. It has an intrinsic capability to handle signal drift and it is much simpler to implement than weighted regression. This calibration method has proved its usefulness for a wide variety of instruments and methods through routine use at the authors laboratories over many years, producing many thousands of results.



**13 Short Course Abstracts****(A1)****INTRODUCTION TO MASS CYTOMETRY***Dmitry R. Bandura, Scott D. Tanner, Vladimir I. Baranov, Olga I. Ornatsky**Fluidigm Canada, Markham, Ontario, Canada**dmitry.bandura@fluidigm.com*

Mass Cytometry, a single cell multiparameter analysis technique based on antibodies labeled with stable isotopes of metals, is turning 7 years old. It is based on the well established technology of inductively coupled plasma mass spectrometry, but in a specific embodiment for single cell detection. Mass Cytometry deals with the analysis of individual particles and distinct ion clouds of sub-millisecond duration, produced in the plasma by the particles.

The short course will focus on the fundamentals of the cell introduction system, ion generation, transport and mass analysis, as well as on the data handling and processing. Use of specially designed elementally embedded polystyrene beads as calibration standards for single particle detection will be discussed. Examples of highly multiparametric single cell analysis by Mass Cytometry using elementally labelled monoclonal antibodies for human leukemia and pancreatic cell lines, bone marrow, peripheral blood and umbilical cord cells, leukemia patient samples will be given. Cell sample preparation aspects for surface and intra-cellular staining, nucleic acids staining and elemental barcoding of cells will be taught. Methodology of calibration and data standardization for quantitation of the antibodies bound to the intracellular or surface antigens will be presented.



**(A-2)**  
**METROLOGICAL APPROCHES FOR QA IN ICPMS**

*Ewa Bulska*

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Quality of chemical measurements is an important issue in today's world influencing quality of life, trade as well as scientific projects. The aim of this short course is to give a comprehensive overview on the importance of the use of metrological principles when performing chemical measurements focusing on ICP MS techniques. The metrological approach towards assuring the quality of the results will be discussed versus the specific requirements of the ISO/IEC 17025 standard.

***AGENDA AND TOPICS:***

- ICP MS: what is measured (counting, signals, concentration)
- Calibration and selection of CRM towards traceability for ICP MS related measurements
- Single laboratory validation of measurement procedures: examples
- Uncertainty of measurement: principles and approaches to evaluation

**(A-3)**  
**TOOLS TO IDENTIFY METAL-CONTAINING MOLECULES: PRACTICAL ASPECTS TO AVOID PITFALLS**

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Despite their low abundance, inorganic elements, such as metals, are essential components of the chemistry of life. Therefore, there is a clear need for tools that can achieve their speciation. Hyphenated techniques with ICP MS instruments as a detector are obviously a good option. However, because of their unstable nature and low abundance, metal containing species are difficult to observe and to identify unambiguously.

This course will focus on different pitfalls that can be faced while analysing metal-containing species and how proper sample preparation, chromatographic or electrophoretic conditions, and elemental and molecular MS data treatment can help to circumvent them.

**(A4)**  
**APPLICATION OF INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION (ICP-AES) SPECTROMETRY – PART I**

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**Abstract**

Inductively Coupled Plasma Atomic Emission (ICP-AES) Spectrometry has become the workhorse in analytical laboratories where multi element analysis has to be performed on a wide range of materials. This short course will review basics of the ICP, a briefly overview sample preparation, aerosol transport and introduction, instrumentation and plasma configurations, spectroscopic and nonspectroscopic interferences.

The course will highlight recent instrumentation developments such as microwave plasmas and critically review benefits and drawbacks of axially vs. radially viewed plasmas using conventional analytical figures of merit such as limits of detection, calibration linearity, and matrix derived nonspectroscopic interferences. Several applications will be described- determination of major, minor and trace elements in geo-environmental, water and waste water samples.

**(B1)**  
**ISOTOPE DILUTION MASS SPECTROMETRY FOR ELEMENTAL ANALYSIS,  
SPECIATION AND METABOLISM STUDIES**

*Jose García Alonso*

The aim of this short course is to provide a comprehensive account of the developments in the field of Isotope Dilution Mass Spectrometry (IDMS) in combination with ICP-MS. The course focuses on the routine implementation of IDMS for total elemental analysis, trace element speciation and metabolism studies with enriched isotopes. In this course we propose an integrated approach that can be employed in any IDMS application. This approach is based on the use of isotope abundances or isotope distributions rather than isotope ratios. We think that this new approach is conceptually simpler and covers all practical applications of IDMS including metabolic and environmental studies.

So, this short course covers the IDMS theory as applied to elemental and speciation analysis including the alternative on-line IDMS together with the work with multiple spikes applied to trace element speciation and metabolism studies. Finally, the concepts of traceability and uncertainty propagation will also be discussed.

**(B2)**

**'UNCERTAINTY BUDGET COOKBOOK - READY TO SERVE IN 2 HOURS'**

*Thomas Prohaska, University of Natural Resources and Life Sciences, Department of Chemistry, VIRIS Laboratory, Konrad*

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*Keywords: Uncertainty budget, error propagation, metrology in chemistry, GUM*

Metrology in chemistry is defined as the science of measurements. The term implements that we do our best in order to fully understand our measurements and to assure the highest quality of our measurements. Even if we do take care about the best measurement results, the validity of these results is determined by their uncertainties, as well, since any measurement is subject to uncertainty. Thus, a proper understanding of uncertainty budgets and the sources of error is crucial for providing proper uncertainty statements but is also an additional prove of the proper understanding of our measurements.

This short course provides the basic understanding for building a proper uncertainty budget. It consists of (a) building the model equation (b) assessing the different sources of uncertainty and (c) propagating the uncertainties to the final results by using the Kragten (spreadsheet) approach. In addition, the Monte Carlo approach for propagating uncertainties will be explained. As final aspect, approaches of propagating errors of depending variables (e.g. calibration curves) will be discussed.

By working on practical examples, the participants should be able to transfer the knowledge to their own laboratories and apply the approaches to their own measurement results.

**(B3)**  
**THE ATMOSPHERIC PRESSURE GLOW DISCHARGE: FUNDAMENTALS AND APPLICATIONS**

*Steven J. Ray*

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This short course will explore the experimental construction, basic spectrochemistry, and analytical utility of the atmospheric pressure glow discharge (APGD). The APGD has experienced significantly increased use in analytical atomic spectroscopy, as well as in applications outside of analytical chemistry. Partially, this increased popularity stems from the simplicity the plasma. The APGD can be sustained with simple structures at atmospheric pressure and requires little ancillary equipment. It is also quite cost-effective, as APGD can often be operated with modest power requirements (e.g. <100W), can often be sustained without any bath gas out in the ambient atmosphere, and are very easily miniaturized. From the analytical perspective the source is also quite attractive; APGD have been successfully used to analyse solid, liquids, and gaseous samples and have been shown to be amendable to optical (absorbance, emission, and fluorescence) and mass spectrometry approaches. The spectrochemistry of the APGD is also intriguing. Many APGD operate in the ill-defined glow-to-arc regime of the GD characteristic curve, meaning that while some aspects of the plasma are certainly indicative of classic glow discharges, other fundamental aspects are better described as flame-like or reminiscent of an arc discharge. Here, the APGD will be described from each of these perspectives: examining the fundamental plasma spectrochemistry of the APGD, reviewing experimental requirements, and examining the APGD as an analytical source in analytical applications. The course will also examine APGD with a view towards miniaturization and portability, in applications that fall outside of typical analytical atomic spectroscopy, as well as potential future applications of these unique plasmas.

**(C1)**  
**SAMPLE PREPARATION IN TRACE ELEMENT ANALYSIS**

*Dirk Wallschläger & Bastian Georg*

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This short course will give a general introduction to the most common sample preparation techniques involved in the analysis of water, soil/sediment/rock or tissue samples by atomic spectrometry. It will be presented at the graduate student level and aimed at the general practitioner in the environmental field without extensive sample preparation experience.

The main focus will be on comparing various complete and partial digestion methods for liquid and solid samples, and discussing under what circumstances using incomplete digestion methods (that do not destroy the entire matrix) is appropriate. In the context of digestion procedures, blanks introduced during sample preparation will be discussed systematically regarding their sources and elimination.

Similarly, leaching/extraction procedures commonly employed to assess bioavailability, mobility or solid state speciation of trace elements will be discussed in terms of performance and compatibility with the measurement techniques.

Also, we will present and discuss strategies for the preconcentration of trace elements from waters and digests/extracts of solid samples, along with methods that isolate the analyte(s) from potential interferences affecting their measurement by plasma spectrometry.

Finally, we will provide an overview of field and laboratory methods for physical fractionation (e.g. filtration) of trace elements in environmental water samples, and discuss appropriate sample preservation strategies for the analysis of total trace element concentrations and trace element speciation in waters.

**(C2)****ICP-MS APPLICATIONS IN EPIGENETIC STUDIES***Katarzyna Wrobel, Kazimierz Wrobel**Chemistry Department, University of Guanajuato, 36000 Guanajuato, Mexico**e-mail: [katarzyn@ugto.mx](mailto:katarzyn@ugto.mx)*

The term epigenetics refers to heritable changes in gene expression that occur without modifications in coding sequence and that are influenced by environmental factors. The most important components on epigenetic machinery involve DNA methylation/demethylation, histone modifications and expression/modifications of noncoding RNAs. Epigenetic modulation of gene expression or silencing is essential during cell differentiation, phenotype development, and also in adaptation and evolution processes; however, undesired aberrations have been associated with pathogenesis of cancer, diabetes and other degenerative diseases. Strong experimental evidence demonstrate that some metals/metalloids and their species are potent epigenetic factors, capable to alter gene expression.

The main focus of metallomics, as an integrated biometal science, is better understanding the specific biological effects of metals/metalloids in biological systems. Since an important aspect of these heteroelements in living organisms is their potential participation in epigenetic events, it has been proposed that such studies would fall within the metallomics domain.

Inductively coupled plasma mass spectrometry (ICP-MS) is a necessary analytical tool in trace element determination, speciation and also in metallomics studies. In addition to exceptional features in terms of low detection limits, multi-elemental and isotopic capabilities, wide dynamic range, high sample throughput, relatively few chemical interferences and easy coupling with different separation techniques, ICP-MS instrumentation enables ultra-trace level determination of few non-metals (P, I, Br, S) that are important in biological systems and also in the environmental studies.

In this short course, several ICP-MS applications of potential epigenetic relevance will be presented, according to the following aspects:

- (i) application of P and Os as natural and external elemental tags, respectively, for the determination of methylation of nucleic acids,
- (ii) analysis of DNA adducts with metal-based drugs,
- (iii) element species as epigenetic factors.



**(C3)****CONTINUOUS FAST SCANNING SINGLE PARTICLE ICP-MS FOR NANOPARTICLE DETECTION AND SIZE DISTRIBUTION DETERMINATIONS: THEORY AND APPLICATIONS***Fadi Abou Shakra and Chady Stephan, PerkinElmer Inc.*

The use of nanoparticles in consumer products is showing a tremendous increase over the previous years. The National Institute of Standards and Technologies reported that nanotechnology-based consumer products are currently entering the market at a rate of 3 to 4 per week and it is estimated that \$2.6 trillion in manufactured goods will contain nanotechnology by 2014[1].

This talk goes over the theory of Single Particle ICP-MS (SP-ICP-MS), the importance of **continuous fast data acquisition** on data quality and describes its use in analyzing metalbased nanoparticles in diver matrices (i.e. environment, food, biological). In this talk we will also show examples of how single particle ICP-MS allows the **differentiation between ionic and particulate signals**, measurement of particle concentration (particles/mL), determination of **particle size and size distribution**. In addition, we will discuss how in well established experiments, it allows users to explore particle **fate**, investigate particle **dissolution kinetics** and study **agglomeration** affinities.

[1] National Institute of Standards and Technologies (NIST), Environmental Leaching of Nanoparticles from Consumer Products <http://www.nist.gov/mml/csd/inorganic/leachnano.cfm> (accessed October 11, 2013).

**(D1)**  
**APPLICATION OF INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION (ICP-AES) SPECTROMETRY – PART II**

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Inductively Coupled Plasma Atomic Emission (ICP-AES) Spectrometry has become the workhorse in analytical laboratories where multi element analysis has to be performed on a wide range of materials. This short course will review basics of the ICP, a briefly overview sample preparation, aerosol transport and introduction, instrumentation and plasma configurations, spectroscopic and nonspectroscopic interferences.

The course will highlight recent instrumentation developments such as microwave plasmas and critically review benefits and drawbacks of axially vs. radially viewed plasmas using conventional analytical figures of merit such as limits of detection, calibration linearity, and matrix derived nonspectroscopic interferences. Several applications will be described- determination of major, minor and trace elements in geo-environmental, water and waste water samples.

**(D2)**

**SYSTEMATIC APPROACH TO ANALYTICAL PROBLEM SOLVING**

*Ramon M. Barnes, University Research Institute for Analytical Chemistry, PO Box 666, Hadley, MA 01035, USA, [barnes@chemistry.umass.edu](mailto:barnes@chemistry.umass.edu)*

This brief course will introduce a system, step-wise approach to solving analytical problems based on the book written by Anderson [1] and a recent chapter on sample preparation [2]. Examples of each step with some illustrations from inductively coupled plasma will be presented.

*[1] R. Anderson, *Sample Pretreatment and Separation*, John Wiley, Chichester. (1991).*

*[2] Ramon M. Barnes, Dário Santos Júnior, Francisco J. Krug, *Introduction to sample preparation for trace element analysis*, in *Microwave-Assisted Sample Preparation for Trace Element Determination*, E.M.M. Flores, ed., Chapter 1, Elsevier, 2014.*

**(D3)****HYPHENATED TECHNIQUES: OVERVIEW ABOUT RECENT TOOLS AND DEVELOPMENTS FOR MARINE ENVIRONMENTAL SPECIATION ANALYSIS***Daniel Pröfrock**Helmholtz-Centre-Geesthacht, Institute of Coastal Research, Max-Planck-Str. 1, 21502 Geesthacht, Germany**e-mail: [Daniel.Proefrock@hzg.de](mailto:Daniel.Proefrock@hzg.de)*

The importance of speciation analysis has been predicted already more than 20 years ago. In parallel the outstanding progress made within this field strongly contributed to the improved understanding of the functional significance and possible interactions of trace elements with ecological compartments and biological systems.

Continuous progress in analytical spectrochemistry in particular within the field of hyphenated techniques has driven the focus of interest from the quantification of small, environmental relevant organometallic compounds (Hg, Pb, Sn) to the separation and identification of partly unknown biological relevant molecules containing trace elements (e.g. Proteins), metalloids (Selenoproteins) or post-translational modifications.

Various new strategies and analytical tools for the complementary application of elemental mass spectrometry (e.g. inductively coupled plasma mass spectrometry (ICP-MS)) which is still generally known as only a “metal” detector beside techniques such as ESI-MS and MALDI-TOF MS and their utilization for new, “non-traditional” application areas have been developed over the years. Within this context the experiences made from the application of proteomic techniques in the field of human diagnostics indicate some interesting possibilities also for the ecological assessment of hazardous substances in particular within the marine environment.

This short course will provide a critical overview about latest developments as well as practical issues related with the most important hyphenation approaches based on HPLC, GC or CE coupled on line with ICP-MS and their recent application for the accurate quantification of contaminants at trace levels as well as the identification and quantification of selected biomarkers. Also interface related issues will be discussed in detail, which are of great importance for the successful application of hyphenated techniques.

**14 Poster Abstracts****(P-1)****BORON SPECIATION AND ACCURATE BORON QUANTIFICATION IN METALLURGICAL GRADE SILICON USING HPLC-ICP-MS AND ICP-OES.***Patrick Galler<sup>1</sup>, Andrea Raab<sup>2</sup>, Sabine Freitag<sup>2</sup>, Kjell Blandhol<sup>1</sup>, Jörg Feldmann<sup>2</sup>*

<sup>1</sup> Elkem AS, Technology, Central Analytical Laboratory, Fiskaaveien 100, P.O. Box 8040 Vaagsbygd, NO-4675 Kristiansand, Norway. E-mail: patrick.galler@elkem.no

<sup>2</sup> University of Aberdeen, College of Physical Science, Department of Chemistry, Trace Element Speciation Laboratory (TESLA), Meston Walk, Aberdeen AB24 3UE, Scotland, United Kingdom.

The accurate quantification of boron (B) in metallurgical grade silicon (MG-Si) has major implications for the Si industry, especially with respect to the development of solar grade Si materials via low cost, metallurgical process routes. Currently these efforts are hampered by a lack of certified reference materials and intra- as well as inter-laboratory reproducibility.

Parallel investigations of a large number of acidic sample digests on two different ICP-OES showed a consistent bias of results between the different instruments, prompting further investigations by ICP-OES as well as HPLC-ICP-MS. In these investigations it could be shown that B exists as at least two different species in sample digests prepared for ICP-OES. One of these B species was found to cause significant underreporting of B concentrations in investigated samples.

Based on these findings a new sample preparation protocol was developed, leading to elimination of the previously observed bias between different ICP-OES. The new sample preparation protocol was analytically validated using ICP-OES and cross validated by prompt gamma activation analysis (PGAA). The B concentration data obtained by ICP-OES was in good agreement with PGAA results. For NIST 57b our results were in excellent agreement with previously published PGAA data.

**(P-2)****IDENTIFICATION OF MERCURY COMPOUNDS IN GYPSUM SAMPLES FROM A LIGNITE BURNING THERMO POWER PLANT**

*Majda Pavlin<sup>1,3</sup>, Matej Sedlar<sup>2,3</sup>, Milena Horvat<sup>1,3</sup>, Radojko Jaćimović<sup>1</sup>, Arkadij Popovič<sup>1</sup>*

<sup>1</sup>*Jožef Stefan Institute, Department of Environmental Sciences, Ljubljana, Slovenia*

<sup>2</sup>*Esotech, d.d., Velenje, Slovenia*

<sup>3</sup>*Jožef Stefan International Postgraduate School, Ljubljana, Slovenia*

Mercury is present as a trace pollutant in coal. During coal burning, mercury is volatilized and emitted through stack gases into the atmosphere, where it becomes a subject of long-range transport. It is well known that about one third of the global mercury emissions are related to coal burning for energy production. Emission control technology for removal of mercury and other contaminants is therefore installed in such industrial settings. Wet flue gas desulfurization (WFGD) system with forced oxidation of sulphite is very efficient in SO<sub>2</sub> removal and therefore most commonly used technology in modern coal fired power plants. It is known that in these systems elemental mercury (Hg(0)) is not retained due to its low solubility in water, the oxidized mercury, however, is almost completely dissolved and retained in the system. Moreover, it was shown that WFGD chemistry can be modified to enable effective oxidation of Hg(0) by air introduced to oxidize SO<sub>2</sub> to gypsum. As a result, the removal efficiency of Hg in WFGD can reach over 90% of Hg present in flue gas. A significant part of the chemistry is the removal of mercury from the solution by the reactions with the solids produced by desulfurization. As a result, mercury levels in such systems are elevated in the resulting gypsum. The focus of the present work is on the understanding of the partitioning and speciation of mercury in the WGD, particularly in gypsum.

As a case study, a gypsum from a lignite burning Thermo Power Plant (TPP) Šoštanj, Slovenia was studied. Distribution of mercury and multi-elemental analysis in different particle size fractions of gypsum was done. In addition, temperature stability of mercury compounds in gypsum was also studied. In the attempt to use the temperature fractionation for the identification of Hg species present in gypsum two methodologies based on atomic absorption detection and mass spectrometry were introduced and validated.

Identification of mercury compounds present in gypsum sample was done with the help of pure Hg compounds (namely HgS, HgO (red and yellow), HgSe, HgSO<sub>4</sub>, Hg<sub>2</sub>SO<sub>4</sub>, HgF<sub>2</sub>, HgCl<sub>2</sub> and Hg<sub>2</sub>Cl<sub>2</sub>) mixed with pure CaSO<sub>4</sub>·2H<sub>2</sub>O. Furthermore, mercury compounds mixed with pure CaSO<sub>4</sub>·2H<sub>2</sub>O were spiked to the real TPP gypsum samples. It was found that thermograms of TPP gypsum samples fitted best with HgCl<sub>2</sub>, Hg<sub>2</sub>Cl<sub>2</sub>, HgO, Hg<sub>2</sub>SO<sub>4</sub> and HgSO<sub>4</sub>. The work is still in progress, however it has to be mentioned that Hg chemistry at higher temperature in complex matrices is not well understood and further methodological improvements are needed to improve this knowledge. The paper will also critically address the potential analytical artefacts commonly present in approaches based on currently used temperature fractionation methodologies.

**(P-3)**

**MEASUREMENT OF COPPER DIFFUSIVITY IN SILICON BY GLOW DISCHARGE MASS SPECTROMETRY**

*Chiara Modanese, Guilherme Gaspar, Lars Arnberg, Marisa Di Sabatino.*

*Norwegian University of Science and Technology (NTNU), Alfred Getz v. 2B, N-7491 Trondheim, Norway.*

*e-mail: chiara.modanese@ntnu.no*

Copper contamination occurs frequently in silicon for photovoltaic applications due to its very fast diffusion coupled with a low solid solubility at room temperature.

The combination of these properties exerts a challenge on the direct analysis of Cu bulk concentration by sputtering techniques like glow discharge mass spectrometry (GD-MS).

In this work, several n-doped monocrystalline (Czochralski) silicon samples were intentionally contaminated with Cu after solidification, and consequently annealed at 900 °C to ensure a homogeneous distribution of Cu in the bulk of the samples. The samples were quenched after annealing to limit the extent of the diffusion to the surface prior to the GD-MS analyses, which were carried out within few minutes after cooling and consecutively at different time intervals. These intervals were kept as similar as possible among the various samples. Samples with different thicknesses were studied in order to evaluate the impact of the out-diffusion over different ranges. In order to evaluate the contribution from external contamination during the Cu contamination and diffusion process, a blank sample (i.e. no Cu contamination) from the same ingot was annealed and analyzed by GD-MS.

The Cu profiles were measured by high resolution GD-MS operating in a continuous mode, where the depth resolution was set to approximately 0.5 µm, over a total sputtered depth in the order of tens of µm. The temperature of the samples during the GD-MS analyses was also measured in order to evaluate the diffusion. The profiles show a strong impact of out-diffusion from the bulk, which takes place in a short time after the annealing.

The Cu concentration profiles were used to calculate Cu diffusivity in these materials.

**(P-4)****QUANTIFICATION OF TEN TRACE ELEMENTS IN FOODSTUFFS BY ICP-MS***Dubascoux S., Nicolas M., Richoz-Payot J., Poitevin E. & Perring L.**Nestle Research Center, Analytical Sciences Competence Pillar - Minerals & Imaging Group, P.O. Box 44, CH - 1000 Lausanne 26**Email : stephane.dubascoux@rdls.nestle.com*

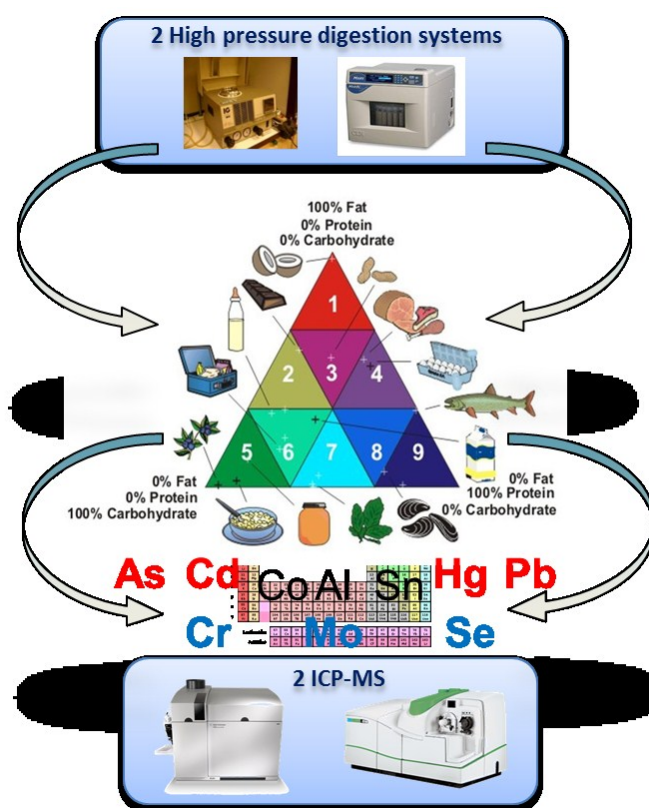
This poster presents performance results of a NESTLE fully validated method for the quantitative determination of ten trace elements (Al, As, Cd, Co, Cr, Hg, Mo, Pb, Se and Sn) in all types of foodstuffs and raw materials including food grade salts by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) after high pressure digestion.

This method is currently used by at least 15 NESTLE regional laboratories worldwide to ensure compliance of finished products and raw materials containing fortifiers (e.g. Cr, Mo and Se) and is applied for the surveillance of their trace contaminant levels (e.g. regulated heavy metals such as As, Cd, Hg and Pb). The goal of this optimization was focused on efficiency (high sample throughput) and simplicity (one-step analysis) for the updated ICP-MS method.

It was validated for one ICP-MS technology (collision cell technology using He as collision gas) on two instruments (Agilent 7700x and Perkin Elmer Nexion 300D) after either High Pressure Ashing (HPA) or Microwave Digestion (MWD). Isopropanol was used as carbon buffer to suppress the so-called “carbon effect” for Se and As determination.

Six different matrices were used for determination of repeatability and recovery and more than 25 various food matrices were used for trueness / recovery assessment in order to fully cover AOAC Food Triangle. Validation results obtained fulfill method performance requirements with trueness / recovery ranging from 70 to 120 %, Practical Limit of Quantification (PLOQ) in finished products at the  $\mu\text{g}/\text{kg}$  level (except for Al with a PLOQ at about  $80 \mu\text{g}/\text{kg}$ ) and expanded uncertainty (U) lower than 50 %.

Details of the present method and comparison of performances will be presented with further objective to be proposed with slight modifications as an internationally recognized method as already realized with the Nestle internal method for nine nutrients (Ca, Cu, Fe, Mg, Mn, P, K, Na, Zn) by ICP-AES which has been officialized as AOAC Official Method Final Action 2011.14.





**(P-5)**

**CADMIUM ISOTOPE RATIO MEASUREMENTS IN ENVIRONMENTAL MATRICES BY MC-ICP-MS**

*Nicola Pallavicini<sup>a,b</sup>, Emma Engström<sup>a,b</sup>, Douglas C. Baxter<sup>b</sup>, and Ilia Rodushkin<sup>a,b</sup>*

<sup>a</sup> *Division of Geosciences, Luleå University of Technology, S-971 87 Luleå, Sweden*

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Various stages of analytical method for high-precision cadmium isotope ratio measurements by MC-ICP-MS (sample preparation, matrix separation, instrumental analysis and data evaluation) were critically evaluated and optimized for analysis of carbon-rich environmental samples. Overall reproducibility of the method was accessed by replicate preparation and Cd isotope ratio measurements in various environmental matrices (soil, sediments, Fe-Mn nodules, sludge, kidney, liver, leaves, mushroom, lichen) and was found to be better than 0.1 ‰ ( $2\sigma$  for  $\delta^{114}\text{Cd}/^{110}\text{Cd}$ ) for the majority of samples. Cd isotope data for several commercially-available reference materials are presented and compared with previously published results where available.

**(P-6)**  
**VARIABILITY IN OSMIUM, LEAD, ZINC AND CADMIUM ISOTOPE  
COMPOSITION IN BIRCH LEAVES FROM SWEDEN**

*Nicola Pallavicini<sup>a,b</sup>, Emma Engström<sup>a,b</sup>, Douglas C. Baxter<sup>b</sup>, and Ilia Rodushkin<sup>a,b</sup>*

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High precision isotope ratio measurements by ICP-SFMS (Os) and MC-ICP-MS were used in pilot study focusing on assessment of factors affecting isotope composition in tree leaves. Summary of results obtained for large number (n>80) of birch (*Betula pubescens*) leaves collected from different locations in Sweden and through entire growing season is presented and potential reasons for observed variability in isotope composition are discussed. Seasonal dynamic of element concentrations and isotope compositions in leaves for Os, Pb, Zn and Cd was compared. With greater understanding observed variations of isotope composition in terms of known processes, it might be possible to identify as yet unknown or unconstrained processes in plants and other biological systems using isotope information.

**(P-7)****AN EXPLORATION OF SAMPLE PREPARATIONS AND DETECTION LIMITS FOR CONDUCTIVE, SEMI-CONDUCTIVE AND NON-CONDUCTIVE MATERIALS AS PERTAINING TO THE NU ASTRUM HR-GDMS.***Dr DeAnn Barnhart<sup>1</sup>, Glyn Churchill<sup>1</sup>, Andrew Burrows<sup>1</sup>, [Bengt Sandgärde<sup>2</sup>](mailto:bengt.sandgarde@msnordic.se)*<sup>1</sup>*Nu Instruments Limited, Unit 74, Clywedog Rd S, Wrexham Industrial Estate, Wrexham, Clwyd UK LL13 9XS.*<sup>2</sup>*Massanalyt Spectrometry Nordic AB, Biskopsvagen 9, SE 12352 Farsta, Sweden**Email: [bengt.sandgarde@msnordic.se](mailto:bengt.sandgarde@msnordic.se)*

High resolution-direct current-glow discharge mass spectrometry (HR-dc-GDMS) is a mature, versatile technique for the direct determination of trace and ultra-trace elements in a variety of materials. Some of the materials which lend themselves to GDMS analysis are predictable such as high purity metals and alloys that have conventional configurations of small blocks with smooth surfaces. However many other types of samples, both conductive and non-conductive of varying sizes, shapes and consistencies may be analysed by GDMS. The work presented here seeks to explore some non-traditional types of samples which may be analysed using the latest generation of HR-dc-GDMS technology, the Astrum from Nu Instruments. Typically GDMS instruments, historically the VG9000 from VG Elemental and currently the Astrum, are fitted with different holders to allow both pin and flat cell capabilities, the pin samples being ~2x2x20mm while the flat samples are ~20x20x10mm, optimally. These different cell geometries allow for a plethora of shapes and sizes of samples to be analysed. If a sample cannot be fashioned into a pin then perhaps it can be pressed onto a binder for analysis using the flat cell method. In this manner, the analysis of such unconventional samples as ball bearings, screws and orthopaedic pins are possible. Thin wires may be analysed by either twisting them into a pin shaped configuration or by cutting them into pieces that can be pressed onto an indium binder. In addition to the versatility afforded by the pin and flat cell geometries, the Astrum has other features which facilitate the analysis of non-conventional samples. Due to the low-flow GD source of the instrument, the Astrum has a sputter rate which can range from 10  $\mu\text{m}$  / 20 minutes for copper to 10  $\mu\text{m}$  / 2 hours for ceramics. This low sputter rate translates into ample time to acquire meaningful and reproducible data for thin and even ultrathin foils and coatings. The low sputter rate also allows for time resolved analysis of coated powders and bulk samples, obtaining information about the contaminants in each matrix layer. Another feature of the Astrum is the cryo-cooled source which allows for both pin and flat cell analysis of low melting point samples such as gallium (m.p. 30°C). Additionally, non-conductive samples can be analysed with the Astrum by using a supporting conductive binder. In this approach, the sputter-redeposition of the secondary electrode material forms a thin conducting layer on the non-conducting sample. Consequently, bombarding particles can penetrate this layer and atomize the non-conducting sample. The most common binder materials are indium and tantalum, due to their high purity and lack of interferences. The indium binder can assume a pin or a flat configuration, both forms allowing for a powder to be pressed onto the indium for analysis. The tantalum binder usually consists of a tantalum pin with a groove fashioned into it which functions to hold the analyte. The different cell geometries, the low sputter rate and the cryo-cooling of the source are among some of the many features of the Astrum which serve to make it a powerful and versatile technique in the determination of trace and ultra-trace contaminants in conductive, semi-conductive and non-conductive bulk materials and powders.

**(P-8)****DETERMINATION OF ESSENTIAL TRACE ELEMENTS OF CASHEW APPLE POWDER FROM VENEZUELA BY INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY**

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*Anacardium occidentale* L. (cashew) is a crop of great interest because the biological properties derived from its chemical composition. Rural people from Zulia region in Venezuela, has focused mostly on the artisanal use of cashew nut, while the pseudofruit or cashew apple is discarded. It is therefore desirable to assess future ingredients which may be obtained from pseudofruits, in order to formulate healthy foods for human consumption. Of great importance for the health are the essential minerals, because they play a key role in the body functions. An alternative to the use of cashew is making cashew apple powder. The aim of the study was to determine the content of sodium (Na), potassium (K), calcium (Ca), magnesium (Mg), iron (Fe), zinc (Zn), copper (Cu), selenium (Se), manganese (Mn) and cobalt (Co) in cashew apple powder. The cashew apple powder was produced experimentally by drying and grinding of pseudofruits creole type (red) which were harvested in growing areas located in the Villa del Rosario de Perija, Zulia State, Venezuela. The pseudofruits were squeezed and dried in an oven for 48 hours at 65°C. Subsequently, the material obtained was ground, sieved and packed in bags for characterization. The concentration of the elements was determined by Inductively Coupled Plasma Mass Spectrometry (ICP-MS). Minerals were found in the following decreasing order of abundance in the cashew apple powder: K>Mg>Ca>Na>Fe>Cu>Zn. Se and Co were not detectable. The mean concentration of the elements was (mg/100g): Na 18.82, 503.64 K, Ca 23.61, Mg 56.66, Fe 3.58, Zn 1.56, Cu 1.67 and Mn 0.66. In conclusion, the cashew apple powder may represent an alternative source of essential minerals for the development and formulation of new products through simple technological processes.

Key words: Essential minerals, cashew apple powder, ICP-MS, food development.

**(P-9)****ESTIMATION OF GAS TEMPERATURE FROM EMISSION OF OH IN MERCURY-CONTAINING CAPILLARY LIGHT SOURCES**

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It is well known that the light source is one of the most important parts in atomic absorption spectrometers. The requirements for the light source in Atomic Absorption Spectrometry are (1) high intensity; (2) homogeneous emission; (3) narrow and not self-reversed line profiles. To satisfy the requirements, light sources undergo optimization; it involves also the search for best working regime. Important part of optimization is the understanding of processes inside the light source. In this study we present results of gas temperature determination in mercury-containing capillary light source using emission of OH.

The light sources under study were high-frequency electrodeless lamps (HFEDLs) filled with mercury and argon (buffer gas pressure 3 Torr). Such lamps are used in LUMEX Zeeman Atomic Absorption spectrometers. HFEDL consists of spherical reservoir (with diameter of 1cm) and capillary part (the length of the capillary was of 2 cm and the discharge size was of 500 $\mu$ m in radius). The discharge was ignited at the capillary part of the lamp. The lamps were capacitatively excited with 250 MHz frequency by means of external electrodes. The emission from lamps was registered using JobinYvon SPEX 1000M spectrometer for three different lamp positions (in respect to Earth's plane): (1) horizontally, (2) vertically with reservoir up and (3) vertically with reservoir down.

When analysing registered spectra, it was observed that, besides the emission of mercury and argon, rotational band of OH at 304.6 nm could be also identified. In many cases rotational temperature determined from OH spectra is a good estimation for gas temperature. In our study we used analysis of relative intensity distribution over rotational lines of OH Q<sub>1</sub> (0-0) branch.

The measurement results for Hg+Ar HFEDLs showed that, when operated vertically with reservoir down, the gas temperature is lower (around 770 K), and in other two positions (horizontally, and vertically with reservoir up) the temperature was estimated to be 830-850 K.

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**(P-10)****DETERMINATION OF GAS TEMPERATURE USING ROTATIONAL SPECTRA OF OH AND C<sub>2</sub> IN THALLIUM-CONTAINING LIGHT SOURCES**

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Rotational spectra of different molecules have been widely used to estimate gas temperature, since these methods are easier and faster than gas temperature determination from Doppler profile of spectral lines. When plasma contains hydrogen molecules, it is very convenient to use H<sub>2</sub> Fulcher- $\alpha$  diagonal branches for temperature determination, however the light sources used for atomic absorption spectroscopy usually do not contain hydrogen, so there is no emission of H<sub>2</sub> molecule. In such cases it is of interest to find other methods for gas temperature estimation.

It has been observed that spectra of high-frequency electrodeless light sources (HFEDLs) can contain emission from different admixtures like, OH, CN, CH or C<sub>2</sub>. They are actually impurities coming from the manufacturing process. Although they are not desirable in light sources, they can be successfully used to estimate gas temperature.

In this work we present results of gas temperature measurements in Thallium-containing electrodeless light sources using rotational spectra of OH (at 304.6 nm) and C<sub>2</sub> (Swan band at 516.5 nm). HFEDLs consist of spherical bulb and a small side-arm made of SiO<sub>2</sub> glass. The lamps were chosen with slightly different fillings: (1) Tl<sup>205</sup>I+Ar (p = 3 Torr) and (2) Tl<sup>205</sup>+SbI<sub>3</sub>+Ar (p = 2,6 Torr). The discharge was ignited by placing the lamp into the electromagnetic field of 100 MHz frequency, and the emission was registered using JobinYvon SPEX 1000M spectrometer. Lamps were operated at different excitation generator voltages (20-29 V), and gas temperature was analyzed in dependence on voltage value.

In spectra of both types it was possible to observe rotational branch of OH radical. Gas temperature determined from OH emission for lamps filled with Tl<sup>205</sup>+SbI<sub>3</sub>+Ar was in range 800-1000K, and for lamps filled with Tl<sup>205</sup>I+Ar obtained temperature values were higher, 900-1100K.

In emission spectra of Tl<sup>205</sup>I+Ar lamps it was possible to identify also C<sub>2</sub> spectrum. Lines of branch (0-0) of Swan band were used to determine gas temperature. Comparison of both temperature values (determined from OH and C<sub>2</sub>) showed that C<sub>2</sub> rotational temperature is 150-200 K higher than the values obtained from OH rotational spectra. It could be explained by the fact that OH forms near the walls of lamp (where temperature is lower) and it is also excited there; however C<sub>2</sub> emission is coming from the central part of the light source, where temperature is higher.

**(P-11)**  
**DETERMINATION OF ZN, CU, PB AND FE IN WINE BY MICROWAVE PLASMA-ATOMIC EMISSION SPECTROMETER**

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The aim of the work was to setup a easy and fast procedure to determine Zn, Cu, Pb and Fe in wine samples by Microwave Plasma-Atomic Emission Spectroscopy (MP-AES). This is a new analytical technique using a nitrogen plasma excited by focused microwave generated via a magnetic field. The maximum temperature reached inside the plasma is 5000 K that creates high intensity atomization emission lines. The most advantages of this technique are: low maintenance and operating costs, ease of use, improvement of the laboratory safety avoiding the use of expensive and hazardous gases, ability to use in the mobile unit and above all it is a multielemental fast instrument.

The wine samples are simple prepared by dilution with distilled water (1:2 for Pb and 1:5 for Zn, Fe and Cu) before being analysed by MP-AES. The analytical wave adopted are Zn 213,857nm, Cu 324,754 nm, Pb 405,781 nm and Fe 371,993 nm.

We analysed 4 wines coming from a Round Robin test (two red wine and two white wine).

Detection limit are 5 times lower than FAAS technique and the recovery for all the elements are between 80-120%.

**(P-12)****DEVELOPMENT AND VALIDATION OF A METHOD FOR DETERMINATION OF 12 ELEMENTS IN BREAST MILK BY ICP-MS QQQ**

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Breast feeding is the recommended source of nutrition for infants in Norway to 6 months of age. About 80% of Norwegian infants are still breast feed at age 6 months, and 43% at the age of 12 months (Meltzer, et al. 2014). Currently the Norwegian Institute of Public Health (NIPH) gives health recommendations to Norwegian women breast feeding based on levels determined in breast milk from Swedish nursing mothers. It is desirable to develop and validate a method that can be used to reassure that the recommendations given are satisfactory for the ten elements cadmium (Cd), calcium (Ca), copper (Cu), iron (Fe), lead (Pb), magnesium (Mg), manganese (Mn), phosphorus (P), selenium (Se) and zink (Zn). Information on iodine (I) and mercury (Hg) in breast milk is scarce. A multi-element method including I and Hg is developed.

In one approach the samples are microwave digested with nitric acid to oxidize and remove the carbon. This digestion has its limitations because the iodine will be oxidized and lost. There is also possible that the mercury will be reduced to Hg(0) and lost from the solution after digestion. To solve the problem with Hg, hydrochloric acid is added after digestion in a final concentration of 2 % (V/V). The chloride complex with Hg and stabilize it in the solution. The second approach is to make an alkaline solution; here the challenge is precipitation of metal hydroxides. To prevent this H<sub>4</sub>EDTA is added to complex the cations. It is also necessary to overcome the carbon effect on the elements with high first ionization energy; for this purpose propan-2-ol is used. In order to break down the cell membranes and keep the proteins in the solution, a non-ionic wetting agent is added, in this case Triton X-100. The advantage with alkaline sample preparation is that iodine determination is easy, and the number of steps is reduced which also reduces the risk of contamination of the samples. Several bases have been tried, to find the optimal solution. Due to limited amount of sample available, time efficiency, and a requirement of a minimum use of reagents and consumables, one common digestion method appropriate for all elements was desirable. The analysis is performed on an Agilent 8800 QQQ ICP-MS in helium collision mode and oxygen reaction mode.

Low quantification limits were required, since the elements Cd, Pb, and Hg are toxic at low levels. In order to avoid any deficiency or excess, it is also important to know the levels of the essential elements. In method development effort was put on lowering the method uncertainty. The validation included the parameters precision, accuracy, linearity, robustness, limit of detection (LD) and limit of quantification (LQ). The best approach all requirements and desires considered will be presented.

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**(P-13)**  
**SEPARATION OF LANTHANOIDES ON REVERSE PHASE COLUMNS MODIFIED WITH VARIOUS LIGANDS: UTILIZING ICP-MS AS DETECTOR**

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Lanthanoides of high purity play a significant role in modern technology. Therefore appropriate separation methods are required which are able to separate them individually to obtain the acquired purity. Also analytical methods are needed to demonstrate the purity. Several chromatographic systems have been used for individual separation of the lanthanoides in an analytical scale including, ion-exchange chromatography, ion-pairing chromatography and extraction chromatography. In most cases UV detection with post-column derivatization has been used, and as this is not a specific detector the identification of the peaks are done with standards of each element.

There are obvious advantages using ICP-MS as a specific detector for metal separation in HPLC. However, many chromatographic separation systems require a mobile phase containing high concentration of buffers and reagents that are not well suited liquids for introduction into the ICP-MS.

In the present work, C18 reverse phase columns were modified with three similar amphiphilic compounds (NST-1, NST-2 and NST-3) according to the procedure given in the work by Dejene et al. [1]. The lipophilic part of the modifier will stick to the C-18 stationary phase and the hydrophilic part is involved in complex formation with lanthanoides. The advantage of these types of stationary phases is that they allow diluted nitric acid to be used as the mobile phase, which is the most suitable liquid to be used for ICP-MS. In addition nitric acid is also very suitable if the system is to be used in a preparative mode.

The impregnated columns were employed to carry out series of elution and optimization tests using standard lanthanoid solutions. Results from the various impregnated columns, i.e. NST-1, NST-2, NST-3 and a mixture of NST-1 and NST-3 will be shown and discussed.

The mixed ligands showed a synergic effect and improved separation of both the lighter and the heavier lanthanoides and eluted them even at lower acid concentration with significantly reduction in retention time. Therefore, mixing of ligands provides an alternative way of tailoring the desired properties for individual separation of lanthanoides.

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**(P-14)****ELIMINATION OF MOLECULAR INTERFERENCES WHEN USING QP-ICP-MS: DETERMINATION OF FIFTEEN RARE EARTH ELEMENTS IN EUROPIUM- AND YTTERBIUM MATRICES UTILIZING ICP-MS AND HPLC-ICP-MS***Viet Hung Nguyen, Dejene Kifle and Grethe Wibetoe**Department of Chemistry, University of Oslo, P.O.Box 1033 Blindern, N-0315 Oslo, Norway**E-mail: [grethe.wibetoe@kjemi.uio.no](mailto:grethe.wibetoe@kjemi.uio.no)*

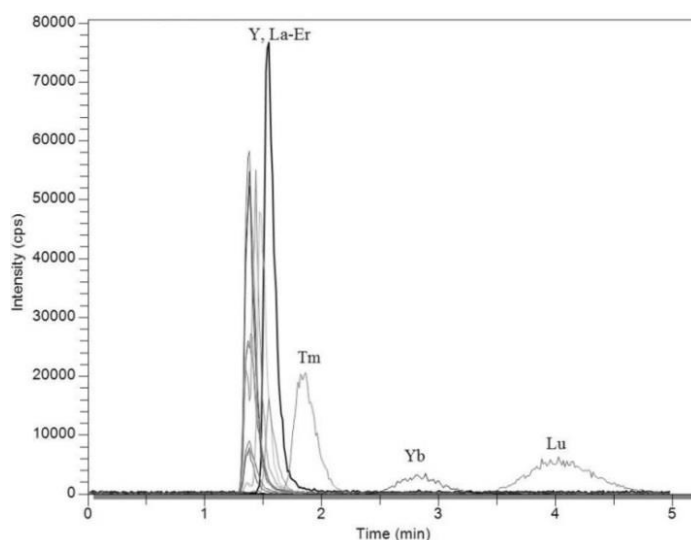
Due to their unique properties, rare earth elements (REEs) are crucial components of many emerging high technological products. For many applications, very high purity of the REEs in the form of metals, oxides etc. are requested. One challenge is to produce the material of the requested high purity; the other is to document it. For the latter case, reliable analytical methods with high detection power to determine the potential impurities are needed.

ICP-MS is one of the most suitable techniques for determination of trace and ultra-trace elements. However, the determination of other REEs at trace level in the presence of high concentration of the matrix REE is not without problems; both suppression effects and spectral interferences need to be considered, where molecular interferences caused by the matrix elements are the most challenging to deal with. For example, as the REEs are among the elements that produce stable oxides and hydroxide ions in the plasma, potential interference will occur for other REE's isotopes with mass 16 and 17 amu higher. Also hydrides of the matrix REE could be formed that will interfere on an element's isotope with mass one amu higher.

The present study utilized both, ICP-MS and HPLC-ICP-MS, to develop methods for determination trace impurities in high-purity Eu and Yb (commercial stock solutions and oxides). It is shown that HPLC-ICP-MS can be used both as a diagnostic tool to identify the contribution of spectral interferences during the method development and for eliminating these interferences for determination of REEs as trace impurities. For both matrices (Eu and Yb) fourteen of the elements could be determined using ICP-MS, only. The spectral interferences from  $^{153}\text{EuO}^+$  on  $^{169}\text{Tm}^+$  (mono-isotopic) and  $^{174}\text{YbH}^+$  on  $^{175}\text{Lu}^+$  in Eu and Yb matrices respectively, required the use of HPLC-ICP-MS to eliminate the spectral interferences. The chromatographic system used is very compatible with ICP-MS as only diluted  $\text{HNO}_3$  is used as mobile phase.

**Figure:** Chromatogram of the REEs using an isocratic program of 5 min with 2.25 M  $\text{HNO}_3$  as mobile phase. The ICP-MS, used at detector was operated at standard operational conditions.

The fig. shows that Eu is separated from Tm (and the other REEs). Eu and Tm are the only REEs that need to be separated before measurement with ICP-MS for determination of other REEs impurities in Eu matrix.



**(P-15)**  
**INVESTIGATION OF ELEMENTAL DISTRIBUTION IN LITHIUM ION BATTERY COMPONENTS USING LA-ICP-MS**

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Laser ablation is a method that allows direct sampling of solids. This is done by evaporating small spots of the sample with a high powered pulsed laser in an argon-helium-atmosphere. The aerosol is transported into an inductively coupled plasma and subsequently analyzed in a mass spectrometer. This approach retains spatial information, within certain limitations and can be used to record three dimensional concentration profiles.

Lithium-ion batteries still experience a loss in performance over time. Although the technology is state of the art in portable consumer electronics, widespread application in automobiles requires improvement beyond the current capabilities. Understanding deterioration processes and mechanisms requires the examination of aged cells. As electrochemical cells do not age homogeneously and electrochemistry in general is heavily dependent on surface properties, bulk analysis is not suitable for investigating these phenomena. LA-ICP-MS promises to be a suitable tool to study these issues.

Our studies show that battery materials are challenging samples that require thorough optimization of the ablation conditions. Due to analysis of the undiluted and undissolved solid sample, laser ablation offers detection limits comparable to liquid analysis with easier sample preparation and most importantly offers spatial information on analyte concentrations.

**(P-16)**

**DETERMINATION OF LITHIUM AND TRANSITION METALS IN LITHIUM-ION BATTERY CELLS BY INDUCTIVELY COUPLED PLASMA – OPTICAL EMISSION SPECTROMETRY AND INDUCTIVELY COUPLED PLASMA – MASS SPECTROMETRY**

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Inductively coupled plasma – optical emission spectrometry (ICP-OES) and inductively coupled plasma – mass spectrometry (ICP-MS) are attractive methods for determination of the element composition of electrode materials. After a microwave digestion and dilution, samples can be directly analyzed.

In this study, both methods were used to investigate lithium and transition metals distribution in lithium-ion battery (LIB) cells. The use and performance of LIBs in electric vehicles is still affected by deterioration over time and lack of high-temperature stability. The performance decline is due to several factors. One important factor is lithium loss. With ICP-MS and ICP-OES, it was possible to analyze the lithium and transition metals content on cathode, anode and separator.

Our studies show that the investigated elements mostly remain on the cathode side. Only a relatively small part of lithium could be determined on the anode side.

**(P-17)****QUANTIFICATION OF PHOSPHOROUS CONTAINING DEGRADATION PRODUCTS IN LiPF<sub>6</sub> BASED ELECTROLYTES WITH IC/ICP-MS***Jennifer Menzel<sup>1\*</sup>, Vadim Kraft<sup>1</sup>, Markus Knepper<sup>2</sup>, Martin Winter<sup>1</sup> und Sascha Nowak<sup>1</sup>*<sup>1</sup>MEET – Batterieforschungszentrum, Corrensstraße 46, 48149 Münster, Germany\*E-Mail: [Jennifer.menzel@uni-muenster.de](mailto:Jennifer.menzel@uni-muenster.de)<sup>2</sup>Deutsche METROHM GmbH & Co. KG, Technisches Büro Nordrhein-Westfalen, Kaninenberghöhe 8, 45136 Essen, Germany

Nowadays lithium-ion batteries (LIBs) are ubiquitous. They supply energy for portable electronic devices like mobile phones, cameras and mp3-players. Their high energy density makes them a promising candidate for the use in electric vehicles (EVs).<sup>[1]</sup> Due to the limitation of fossil fuels this development is significant to make renewable energies usable for individual mobility. Besides the lower driving range in comparison to combustion engines the limited battery lifetime due to aging processes is an important challenge.<sup>[2]</sup>

Many aging processes can be ascribed to reactions of the conducting salt in battery electrolytes<sup>[3]</sup>. For lithium hexafluorophosphate (LiPF<sub>6</sub>), the most commonly used conducting salt many decomposition products are already known.<sup>[2]</sup> Upon reaction with the organic carbonates used as solvent alkyl phosphates are formed, which are suspected to play a role in the formation of the *Solid Electrolyte Interface* (SEI), a protection layer on the anodic surface. The alkyl phosphates were identified with ion chromatography (IC) coupled to electrospray ionization-mass spectrometry (ESI-MS).<sup>[4]</sup> The quantification of these alkyl phosphates can provide knowledge about the kinetics of the decomposition of LiPF<sub>6</sub>. The quantification can not be performed with IC and conductivity detection as the separation from fluoride is insufficient. Due to the absence of standards for these species quantification via IC/ESI-MS is not possible either.

In order to establish a quantification procedure for these substances a method needs to be developed which is selective for the alkyl phosphates. ICP-MS is selective for the phosphorous atoms of the alkyl phosphates and can be detected independently from the fluorine which is eluted at a similar elution time. For the separation of the different alkyl phosphate species a two dimensional IC is used. After the first separation of the alkyl phosphates from some fluorophosphate species, the alkyl phosphates are separated on a second high capacity column and then forwarded into the ICP-MS. In order to enable a reliable quantification an internal tellurium standard is used.

Upon investigations of a commercially available electrolyte after the aging process, different alkyl phosphate species have been separated and identified. The quantification gave an amount of the alkyl phosphates in the lower ppm range.

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**(P-18)**  
**QUANTITATIVE DETERMINATION OF AGING RELATED CHANGES IN THE LITHIUM DISTRIBUTION IN LFP ELECTRODES.**

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Due to a lack of reliable methods for  $\mu\text{m}$  scale determination of the lithium distribution in aged LIB electrodes we developed a method using LA-ICP-MS. The solid sample is investigated using a 213 nm Laser with spot sizes ranging from 10-200  $\mu\text{m}$  and subsequent analysis in an ICP-MS. Evaporating small parts of the sample with the laser and analyzing the elemental composition of each spot allows a three dimensional investigation of the electrode composition.

Even state of the art LIB systems show some degree of performance loss over time. Various causes for this have been proposed or identified. The distribution of lithium in the LIB is one of the major factors concerning the performance. Thus changes in the distribution can have a huge impact on the cycle and calendar life of cells. The method we developed could be used to quantitatively study the lithium content in the single digit  $\mu\text{m}$ -range.

**(P-19)**  
**SPECIATION ANALYSIS OF ANTIMONY IN DIFFUSIVE GRADIENTS IN THIN FILMS (DGT) BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY - ISOTOPIC DILUTION - INDUCTIVELY COUPLED PLASMA MASS SPECTROMETER (HPLC-ID-ICP-MS)**

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Antimony (Sb) is considered to be a nonessential element in plants, animals or humans. The most common oxidation states are +5, +3, and -3. It exists mainly as Sb(III) and Sb(V) in environmental, biological, and geochemical samples.

The technique of DGT is designed to measure in situ labile metals. Inorganic species of antimony (Sb) in aqueous solutions are trapped in a binding Fe-oxide resin gel after their diffusion across diffusive layer. We extracted and preserved Sb species from the resin with EDTA solution before performing the analysis by HPLC-ID-ICP-MS with anionic exchange column.

Here, we report for the first time detailed performance characteristics of the Fe-oxide gel and associated DGT devices obtained by deployment in known solutions of trivalent and pentavalent Sb. The diffusion coefficients for each chemical forms were determined using an independent diffusion cell. Theoretical responses were obtained with studies in different ionic strength (from deionized water to 0.1 M NaNO<sub>3</sub>) and pH (5-9). The potential capability for simultaneously measuring labile forms of antimony in acidic to neutral, fresh to brackish waters was demonstrated. New data on the diffusion coefficients of Sb(V) and the first data on diffusion coefficient for Sb(III) in the diffusive gels are provided to allow accurate calculation of DGT measured concentrations.

Speciation analysis of Sb(III) and Sb(V) in aqueous samples was performed through extraction from DGT resin after deployment and on-line isotope dilution concentration determination after a chromatographic separation. The separation of Sb(III) and Sb(V) was achieved using an anion exchange column and 10 mmol L<sup>-1</sup> EDTA and 1 mmol L<sup>-1</sup> phthalic acid at pH 4.5 as a mobile phase. The eluent from the HPLC was mixed with an enriched <sup>123</sup>Sb spike solution that was pumped by a peristaltic pump with a constant flow rate (0.04 mL min<sup>-1</sup>) in a three-way valve.

The combination of HPLC-ID-ICP-MS, as analytical technique, Fe-oxide DGT, as adsorption system, and EDTA, for preservation of different species represent an excellent point of departure for antimony investigation in environmental, especially in groundwater.

**(P-20)****SERUM LEVELS OF ESSENTIAL MINERALS IN NURSE STUDENTS WITH SMOKING HABITS FROM THE ZULIA UNIVERSITY, VENEZUELA**

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The addiction to tobacco is a critical aspect that has generated much interest today in the health area staff. But the relationship between cigarette smoking and mineral content in the body is a little studied aspect and inconsistent results nationally and internationally. The objective of the research was to determine the effects of smoking on the serum levels of essential minerals in nursing students at the University of Zulia. This study was correlational with cross-sectional design. The sample was represented by 56 students of different genders, aged 18-30 years. A questionnaire previously validated by experts was applied to assess the prevalence, knowledge and attitudes towards smoking. The iron (Fe), zinc (Zn) and copper (Cu) in blood serum was quantified by Inductively Coupled Plasma Mass Spectrometry (ICP-MS). The Spearman correlation analysis was applied to relate smoking to levels of minerals. A high prevalence of smoking habit (50.9%) was found among nursing students, and 26.3% started smoking during the nurse studies. The 17.5% smoked in college, 14.0% of current smokers cigarettes consumed daily. The 42.1% of smokers want to leave that habit, and 66.7% supported completely suppress smoking in hospitals or health centers. Zn levels were significantly higher ( $p < 0.05$ ) in student smokers ( $0.50 \pm 0.08$  mg/L) compared with nonsmokers ( $0.45 \pm 0.10$  mg/L). A significant correlation ( $r = 0.31$ ,  $p < 0.05$ ) between serum Zn and smoking was found in Nursing student from the University of Zulia.

Key words: Essential minerals, nursing student, ICP-MS, smoking habit.



**(P-21)****ANALYTICALLY MONITORING THE EFFECT OF FRACTURING ACTIVITY**

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Fracking, a process used by the petrochemical industry to increase extraction efficiency of natural gas that is trapped in bedrock is under scrutiny due to potential hazard to the environment. The process of hydraulic fracturing uses a water/chemical/sand mixture pumped at high pressure to induce fracturing of the rocks and release trapped fuels. It is a tactic that has been used in a large portion of wells over the last decades, but recent refinements to the process have increased its use to exploit energy resources that had previously been inaccessible. In some countries, fracking is banned and in the US, the State of California has recently signed new legislation, effective in 2014 that will require groundwater and air quality monitoring.

The fracking process results in addition of chemicals to the subsurface along with mobilization of anions, cations, metals, and radioisotopes in the shale layers that are returned to surface as flowback waters. Analytical instrumentation is required to determine the concentrations of these analytes so that environmental impact of groundwaters can be minimized, fracking processes can be improved and wastewater or brines can be characterized prior to disposal.

This poster presents how questions surrounding the impact of fracking can be answered using several different types of analytical instrumentation. Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) and Inductively Coupled Plasma Mass Spectrometry (ICP-MS) methods have been developed according to EPA methods 200.7 and 200.8 respectively and are here presented.

**(P-22)****DISSOLUTION STUDIES OF SPARINGLY SOLUBLE ThO<sub>2</sub> WITH HR-ICP-MS***Emmi Myllykylä<sup>1</sup>, Tiina Lavonen<sup>1</sup> and Kaija Ollila<sup>1</sup>*<sup>1</sup> VTT Technical Research Centre of Finland, P.O. Box 1000, FI-02044 VTT (emmi.myllykyla@vtt.fi)

The objective of this study was to investigate the solubility and initial dissolution rate of crystalline ThO<sub>2</sub>. The research was performed as a part of the EU project called REDUPP, which aimed at better understanding of the dissolution processes of the oxides, ThO<sub>2</sub>, CeO<sub>2</sub>, and UO<sub>2</sub>, which are isostructural with fluorite, the structure of UO<sub>2</sub> matrix of spent nuclear fuel.

In literature, the solubility values of ThO<sub>2</sub>, as well as the hydrolysis constants of thorium show great discrepancies [1]. The main reasons for the discrepancies are the tendency of Th to undergo polynucleation and colloid formation, its strong absorption to surfaces, and the low solubility of both Th<sup>4+</sup> hydroxide and hydrous oxide. The solubility values have been observed to depend on the crystallinity of ThO<sub>2</sub>, but the surface phenomena of the oxide may also play a role in the dissolution.

Prior to the experiments, the sintered ThO<sub>2</sub> pellets were made to meet an ideal composition and microstructure similar to the fluorite structure of UO<sub>2</sub> fuel. Making of the pellets is described in detail elsewhere [2]. The surface analyses, performed prior to the solubility experiments, showed a microstructure similar to UO<sub>2</sub> fuel pellets; randomly oriented crystals with grain size from 10 to 30 μm.

The dissolution experiments were conducted with two particle sizes, 60 to 180 μm and 2 to 4 mm in 0.1 M NaCl or 0.01 M HNO<sub>3</sub> in an Ar glove-box. Another series of experiments was conducted in 0.01 M NaCl solution using <sup>229</sup>Th spike to study reversibility of surface reactions. These experiments were conducted either with single solid pellet or 2 to 4 mm particles. The concentration of <sup>232</sup>Th and the isotopic ratio of <sup>229</sup>Th/<sup>232</sup>Th in the nonfiltered and ultrafiltered samples were analysed with HR-ICP-MS (Element 2 by ThermoScientific). The analyses were performed using standard solutions with known concentrations diluted from AccuTrace<sup>TM</sup> Reference Standard SQS-01. A control sample for the analysis was prepared from standard CLMS-1 solution by SPEX. In addition, all the blank, standard and control samples contained a known amount of an internal standard, indium. Analyses of Th were performed by using low resolution (R ≈ 300). The detection limit for thorium was between 1 · 10<sup>-12</sup> mol/L and 4 · 10<sup>-12</sup> mol/L depending on the solution matrix and daily efficiency of the instrument.

In the experiments, the concentration level of Th (≥ 10<sup>-9</sup> mol/L) at pH ≤ 4 was a bit lower than the theoretical solubility of crystalline ThO<sub>2</sub>. At higher pH values Th concentration is dominated by the sparingly soluble hydrolyzed Th phases, and HR-ICP-MS enabled the low concentration of Th to be analyzed down to 10<sup>-12</sup> mol/L. The results of the experiments show that for ThO<sub>2</sub> the process of dissolution/precipitation (sorption?) occurs simultaneously and demonstrate reversibility.

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[2] REDUPP Deliverable 1.1 (found on the REDUPP web page, [www.skb.se/REDUPP](http://www.skb.se/REDUPP))

**(P-23)****EFFECTS OF SOME HEAVY METALS ON THE RHIZOBACTERIUM AZOSPIRILLUM BRASILENSE: SPECTROCHEMICAL ANALYSES OF METAL UPTAKE AND METABOLIC RESPONSES***Alexander A. Kamnev, Anna V. Tugarova**Institute of Biochemistry and Physiology of Plants and Microorganisms, Russian Academy of Sciences, 13 Prospekt Entuziastov, 410049 Saratov, Russia**E-mail: aakamnev@ibppm.sgu.ru, a.a.kamnev@mail.ru*

Rhizobacteria of the genus *Azospirillum*, and particularly its most widely studied ubiquitous species *A. brasilense*, are of agrobiotechnological interest because of a number of their beneficial traits including phyto-stimulating capabilities. *A. brasilense* is also a good model for studying microbial ecology, as it includes strains which occupy different ecological niches in the plant rhizosphere. Thus, its wild-type strain Sp245 is a facultative endophyte (capable of penetrating into the plant root tissues), while strain Sp7 is an epiphyte (colonizing exclusively the root surface), and some of their metabolic responses to similar stress factors have been documented to differ [1–3].

In this work, uptake of each of several metals ( $\text{Co}^{2+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Zn}^{2+}$ , present in the culture medium as model contaminants at 0.2 mM) by live cells of *A. brasilense* strains Sp7 and Sp245, as well as their concentrations in cells as natural trace elements were analysed using ICP-MS (Table 1). Metabolic responses of the two strains to the heavy metals were studied by Fourier transform infrared (FTIR) spectroscopic analyses of dry cell biomass, while  $\text{Co}^{2+}$  binding and transformations in live cells were monitored by  $^{57}\text{Co}$  emission Mössbauer spectroscopy (EMS) [1, 3, 4] in rapidly frozen cell suspensions. Despite comparable metal uptake levels, the strains responded differently to each metal with regard to poly-3-hydroxybutyrate (storage biopolyester) accumulation. EMS data also showed differences in  $^{57}\text{Co}^{2+}$  metabolic transformations by the two strains, in line with their different ecological behaviour, in particular, under heavy-metal stress [1–3].

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**Table 1.** Content of metals in cell samples of *A. brasilense* (strains Sp7 and Sp245)<sup>a</sup>

Cultivation medium	Metal content (µg per gram of dry cells) analysed by ICP-MS					
	Co		Cu		Zn	
	Sp7	Sp245	Sp7	Sp245	Sp7	Sp245
Standard (control)	0.5	3.1	47	21	25	41
With 0.2 mM $\text{Co}^{2+}$	<b>118</b>	<b>134</b>	2	33	27	58
With 0.2 mM $\text{Cu}^{2+}$	0.7	0.8	<b>477</b>	<b>438</b>	35	53
With 0.2 mM $\text{Zn}^{2+}$	0.6	1.6	7	33	<b>4240</b>	<b>2140</b>

<sup>a</sup> The metals were either present in the standard cultivation medium as impurities (data given in normal font) or added to the medium up to 0.2 mM (data given in **bold** font).

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**(P-24)****NEW DGT ADSORBENTS FOR DISSOLVED INORGANIC AND ORGANIC PHOSPHORUS COMPOUNDS IN WATER (DIP AND DOP)***Oddvar Røyset<sup>1</sup>, Tomas Adler Blakset<sup>1</sup>, Rolf Vogt<sup>2</sup>, Christian Mohr<sup>2</sup>, Neha Parekh<sup>2</sup>,**1) Norwegian Institute for Water Research (NIVA), Gaustadalléen 21, N-0349 OSLO, +47 90149541, [oddvar.roeyset@niva.no](mailto:oddvar.roeyset@niva.no), 2) Department of Chemistry, University of Oslo, 0315 OSLO, Norway.*

The Ferrihydrite (iron hydroxide) and Metsorb™ (titanium oxide/hydroxide) adsorbents were studied in the Research Council of Norway project EUTROPIA (Rolf Vogt, UIO) from 2009 to 2012. Both adsorbents collected dissolved inorganic phosphorus compounds (DIP mainly orthophosphate species) with high efficiency. The diffusion coefficients (D) for the orthophosphate species decreases with increasing pH due to the larger negative charge with pH (i.e.  $\text{H}_2\text{PO}_4^-$  with charge -1 from pH 3 to 7 and  $\text{HPO}_4^{2-}$  with charge -2 from pH 7-11). A new pH correction function was developed where D decreases with 0.15 units per pH. In a field study at Lake Vansjø this correction improved the agreement between the results by DGT-DIP fractions and grab sample DIP fractions.

The dissolved organic phosphorus (DOP) fraction is a heterogeneous compound group from typical low molecular weight (<1000 Da) phosphorus containing sugars, inositols (phytic acid derivatives), lipids, nucleotides etc, to high molecular compounds (Mw 1000 to 10000 Da) of the Dissolved Natural Organic Matter group (DNOM/NOM). Both adsorbents collected the DOP fraction with high efficiency. A laboratory study showed high collection efficiency for two low molecular test compounds (adenosine monophosphate (AMP Mw 347) and phytic acid Mw 736). The elution efficiency was high and the sampling precision was good. The molecular weights of the DOP fraction in natural water range from 200 to at least 10000 Da with D values from 4.5 to 1.4  $10^{-6}$  cm<sup>2</sup>/sec. Thus one single D value cannot be assigned to cover whole this Mw range. In the runoff from agricultural areas at Lake Vansjø, the results for the DGT-DOP fractions and grab sample DOP fractions, agreed best when using D values between 4.0 and 3.5  $10^{-6}$  cm<sup>2</sup>/sec. This indicates that the DOP fraction is mainly low molecular in the range 300 to 2000 Da, agree with results from agricultural areas (Røyset et al. 2014), but requires more research to verify if this is a general pattern in agricultural runoff.

A new carbon, nitrogen, phosphorus analyzer has been developed at NIVA for the determination of up to 7 different CNP nutrient fractions, including the phosphorus fractions collected by DGTs. The DGTs with Ferrihydrite and Metsorb adsorbent are new sampling and analysis tools of the DIP and DOP fractions in water. More information can be found in workshop presentation at the project website and in the project report (Røyset et al. 2014).

<http://www.mn.uio.no/kjemi/english/research/projects/eutropia/index.html>

Røyset, O, Vogt, RD, Mohr, CW, Parekh, NA (2014) Sampling of dissolved inorganic and dissolved organic phosphorus compounds in natural water by Diffusive Gradients in Thin-films (DGT), NIVA report 6590-2013, Norwegian Institute for Water Research, Oslo.

**(P-25)****DGT-PROBES APPLIED FOR DETERMINATION OF METAL DISTRIBUTION AND METAL EXCHANGE ACROSS THE SEDIMENT-WATER INTERFACE IN SØRFJORDEN, NORWAY.***Morten Schaanning, Anders Ruus, Sigurd Øxnevad and Oddvar Røyset.**Norwegian Institute for Water Research (NIVA), Gaustadalléen 21, N-0349 OSLO, Norway,  
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Sediments in the inner part of the fjord is heavily contaminated from 20th century industrial discharges. As part of the development of a remediation plan for the area, metal fluxes were to be determined in six selected areas. In five areas this was done by incubation of triplicate box-core samples in a mesocosm laboratory and measuring the difference in metal concentrations in the water flowing in and out from each box. In addition the metal distribution in pore water was determined on 15 cm DGT-probes sliced in 5-20 mm depth intervals after 24h exposure in the box-core samples. In the sixth sub-area, however, box-core samples could not be drawn due to a geotextile and 50 cm sand layer placed in 1992 to reduce metal leakage to the overlying water. Therefore, DGT probes were deployed *in situ* by divers, retrieved, sliced and analyzed in the same way as was done with the probes deployed in the box-core samples in the mesocosm. The fluxes determined in the box-core samples correlated well with the metal uptake on the DGT-probes and the functions derived from linear regression for each metal could then be used for calculation of the fluxes from the capped sediments in the sixth area. The DGT-profiles also revealed differences between the cap material and the more fine-grained natural sediments, possibly related to cap function.

Ruus, A., Schaanning, M.T., Iversen, E.R., Øxnevad, S. & Røyset, O. 2013. Kvantifisering av tungmetalltilførsler i indre del av Sørfjorden, Hardanger. NIVA rapport 6453-2012. Norwegian Institute for Water Research, Norway

**(P-26)**

**BENEFITS OF TRANSITIONING FROM FAAS TO THE AGILENT 4200 MP-AES**

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Reducing ongoing operating costs, increasing safety, improving analytical performance, and ease of use are some of the key challenges facing current users of Flame Atomic Absorption Spectrometry (FAAS). With the introduction of the Agilent Microwave Plasma-Atomic Emission Spectrometer (MP-AES), these challenges have been overcome, making it the ideal instrument for laboratories looking to transition away from FAAS to a more powerful, less expensive and safer technique. Additionally, with the extra performance of the MP-AES, the sample preparation process can also be significantly simplified, saving time and money. The second generation Agilent 4200 MP-AES features an improved waveguide design and updated torch that is capable of running samples with high total dissolved solids without compromising detection limits.

**(P-27)**

**SINGLE PARTICLE ANALYSIS OF NANOMATERIALS WITH AGILENT 7900 ICP-MS**

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Nanoparticles (NPs) are defined as ultrafine particles with one dimension between 1 –100 nm. But while their use is constantly increasing, questions and concerns have been raised about their safety and their health impact. For this reason, there is an urgent need to develop analytical methods that are suitable for the particular evaluation of NPs.

An interesting approach for the characterization of NPs has been developed by Degueldre et al. using ICP-MS. If samples containing NPs are introduced at a low number of particles in solution, analysis in time resolved mode makes possible to collect the intensity for a single particle as it is vaporized and atomized in the plasma. Then, each measured data point can be correlated to the size and mass fraction of a unique NP. This method of NP characterization is called Single Particle ICP-MS (SP-ICP-MS) analysis. In the present work the analysis of silver NPs has been carried out using the Agilent 7900 in SP-ICP-MS mode. The method allows the determination of the nanoparticles mass concentration but also the evaluation of their median size and size distribution.

**(P-28)**

## **BIOAVAILABILITY OF CADMIUM FROM LINSEED AND COCOA**

*Max Hansen, Rie R. Rasmussen and Jens J. Sloth*

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The exposure of the European population to cadmium from food is high compared with the tolerable weekly intake of 2.5 µg/kg bodyweight set by EFSA in 2009. Only few studies on the bioavailability of cadmium from different food sources has been performed but this information is very important for the food authorities in order to give correct advises to the population.

The aim of this study was to investigate the bioavailability of cadmium from whole linseed, crushed linseed, cocoa and cadmium chloride in rats.

An experiment where 40 rats were divided into 4 groups and a control group and dosed with whole linseed, crushed linseed, cocoa and CdCl<sub>2</sub> for 3 weeks was performed. Linseed or cocoa made up 10% of the feed (by weight) and was added as a replacement for carbohydrate source. The rats were dosed for 3 weeks and the cadmium content in the rats' kidneys was measured by ICPMS as a biomarker for the exposure during the whole life. Efforts were made to keep unintended exposure as low as possible and the cadmium content was measured in whole feed and all individual feed components.

The total intake of cadmium during the lifetime of the rats was calculated and the percentage of the cadmium which could be measured in the kidney compared to the calculated total intake was as follows: Control 2.0 %, Crushed linseed 0.9 %, whole linseed, 1.5 %, cocoa 0.7 % and CdCl<sub>2</sub> 4.6 %.

Based on this study it could not be concluded that the bioavailability in rats from whole linseed is lower than for crushed linseed. It was concluded that the bioavailability of cadmium from cocoa was similar or maybe a little lower than the bioavailability of cadmium from linseed.



**(P-29)****DETERMINATION OF IODINE AND IODINE COMPOUNDS IN MARINE SAMPLES BY ICPMS AND HPLC-ICPMS***Maiken S. Hansen, Daniel J. Lewandowski, Rie R. Rasmussen, Birgitte K. Herbst and Jens J. Sloth**National Food Institute (DTU Food), Division of Food Chemistry – Technical University of Denmark, Søborg, Denmark*

By now it is a well-known fact that iodine is an essential trace element for the growth and development of the human body. Because of iodine deficiency, some countries have added iodate to salt in order to increase the iodine intake. However, some people prefer iodine from more natural sources like seaweed and fish, which contain elevated levels of iodine (fish typically 1-10 mg/kg and seaweed up to 8000 mg/kg). These marine food items may contain different iodine species, which may have different bioavailability and toxicity, and hence there is an increased interest in developing analytical methods for determining the different iodine species.

For determining the total iodine concentration in marine samples five different extraction methods were compared. The most efficient and precise method was then used for determining the total concentration of iodine in seaweed and fish samples using inductively coupled plasma mass spectrometry (ICPMS).

Furthermore 32 marine samples were analyzed for contents of iodide, iodate, moniodotyrosine (MIT) and diiodotyrosine (DIT). The samples were extracted using the enzyme pancreatin followed by analysis with reversed phase high performance liquid chromatography (HPLC) coupled to ICPMS.

These studies may be a stepping stone for further studies that can clarify the cycle and implications of iodine species in relation to the use of marine food items as iodine sources.

**(P-30)****STRATEGIES FOR THE MEASUREMENT OF LEAD ISOTOPE RATIOS IN RADIOGENIC MINERALS BY LASER ABLATION MULTICollector ICP-MS***A. Reguera Galan, M. Moldovan and J.I. Garcia Alonso**Department of Physical and Analytical Chemistry, Faculty of Chemistry, University of Oviedo. Julian Claveria 8, 33006 Oviedo, Spain.**e-mail: jiga@uniovi.es*

Provenance studies of copper and bronze archaeological artefacts can be performed by comparing lead isotope ratios both in artefacts and metal ores collected in the copper mines. During our study of lead isotope ratios in ancient copper mines in the north-west of Spain<sup>1-3</sup> we have found highly radiogenic lead in La Profunda mine where uranium containing minerals (such as zeunerite, an uranium copper arsenate) were described. Lead isotope ratios 207/206 between 0.2 and 0.8 were measured in 30 samples analysed after dissolving the minerals and separating lead by chelation chromatography.<sup>3</sup> In order to gain more information about this very special deposit we have applied laser ablation ICP-MS to several mineral samples obtained from La Profunda mine including azurites, malachites and zeunerites.

The experimental procedure consisted in the on-line nebulisation of a thallium solution for internal mass bias correction and single point analysis by Laser Ablation. Lead isotope ratios were determined by linear regression plotting the signals obtained at one mass against the signal obtained at the other masses during the whole ablation process. It was observed that the lead concentrations and isotope ratios were highly heterogeneous within the minerals and even within a single point laser sampling requiring the development of alternative strategies for data treatment. The lead isotope ratios were, in many instances, a mixture of, at least, two very similar but different components. We developed a procedure to separate the two components based on the residuals of the linear regression for the 207/206 and 208/206 isotope ratios with satisfactory results. It was also observed that, for the zeunerite samples, the isotope ratio for thallium changed during the ablation of the mineral indicating matrix effects affecting mass bias. These effects were not observed with the azurite and malachite samples and could be ascribed to the presence of the high concentration of uranium in zeunerite.

So, it was possible to characterise the three minerals in terms of lead isotope ratios and to obtain "isoscares" for some of the samples which could be compared to metal distributions obtained by SEM. All three minerals contained highly radiogenic lead being the malachite the most radiogenic sample with 206/204 isotope ratios approaching 700.

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- ✓ Single og multi-element
- ✓ 125 ml og 250 ml
- ✓ 1.000 og 10.000 µg/ml
- ✓ Anioner og kationer

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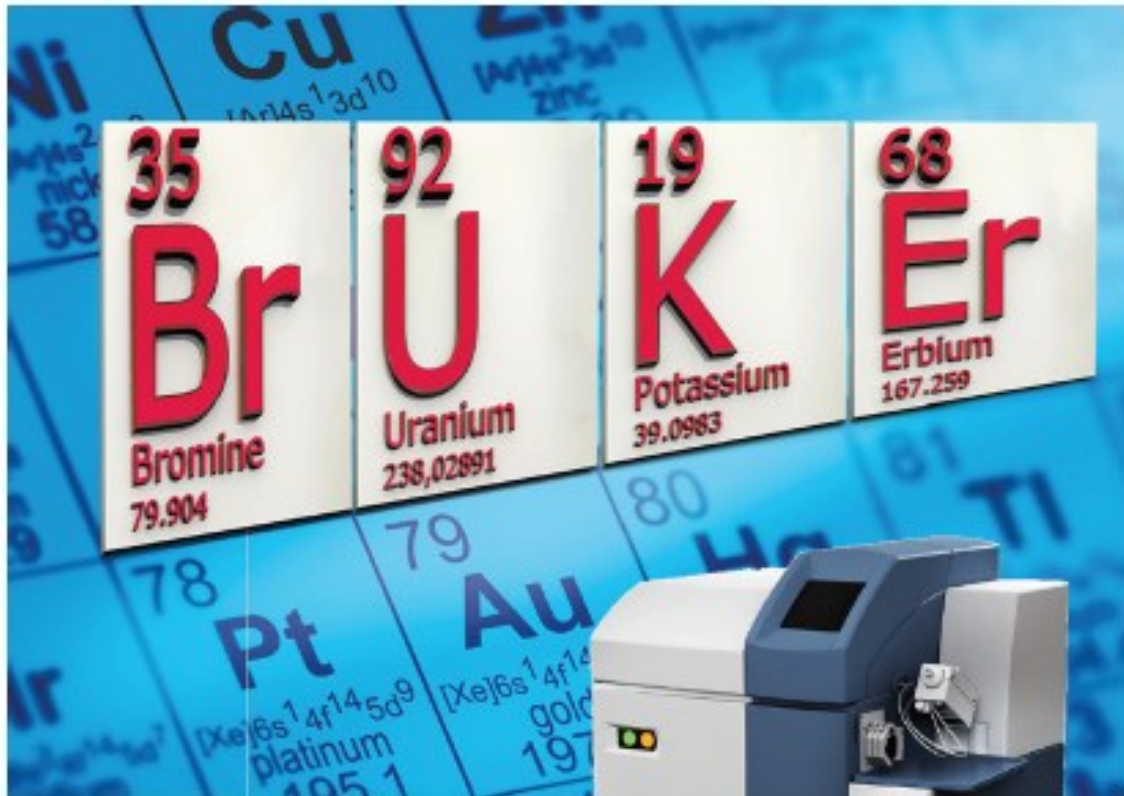
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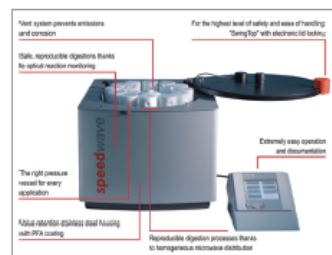
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ICP-MS



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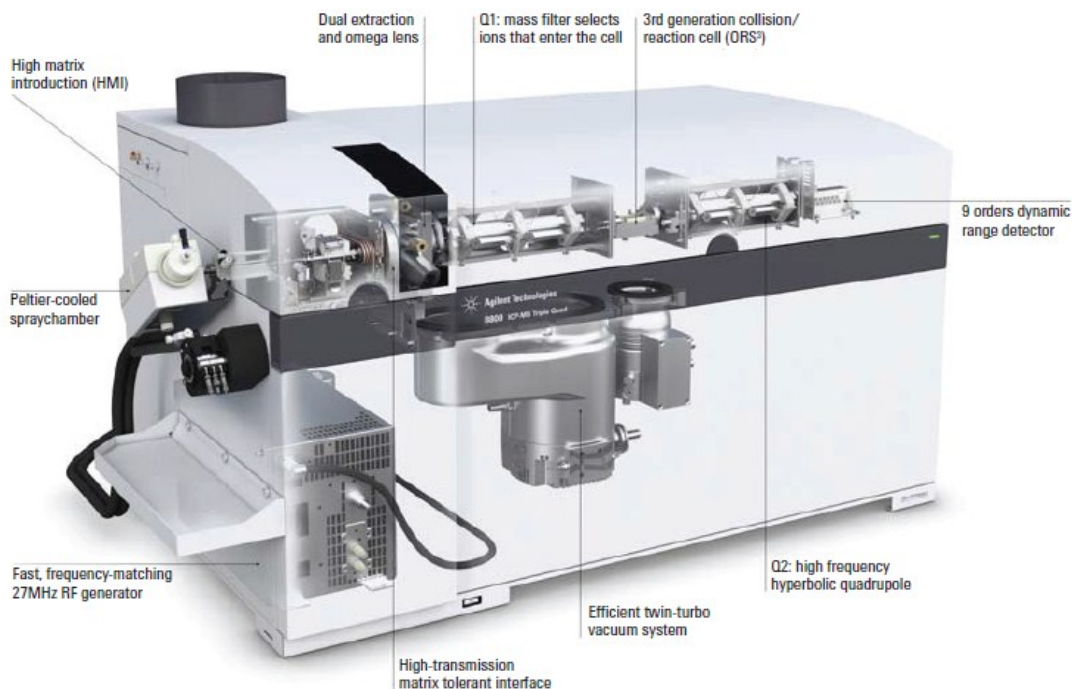
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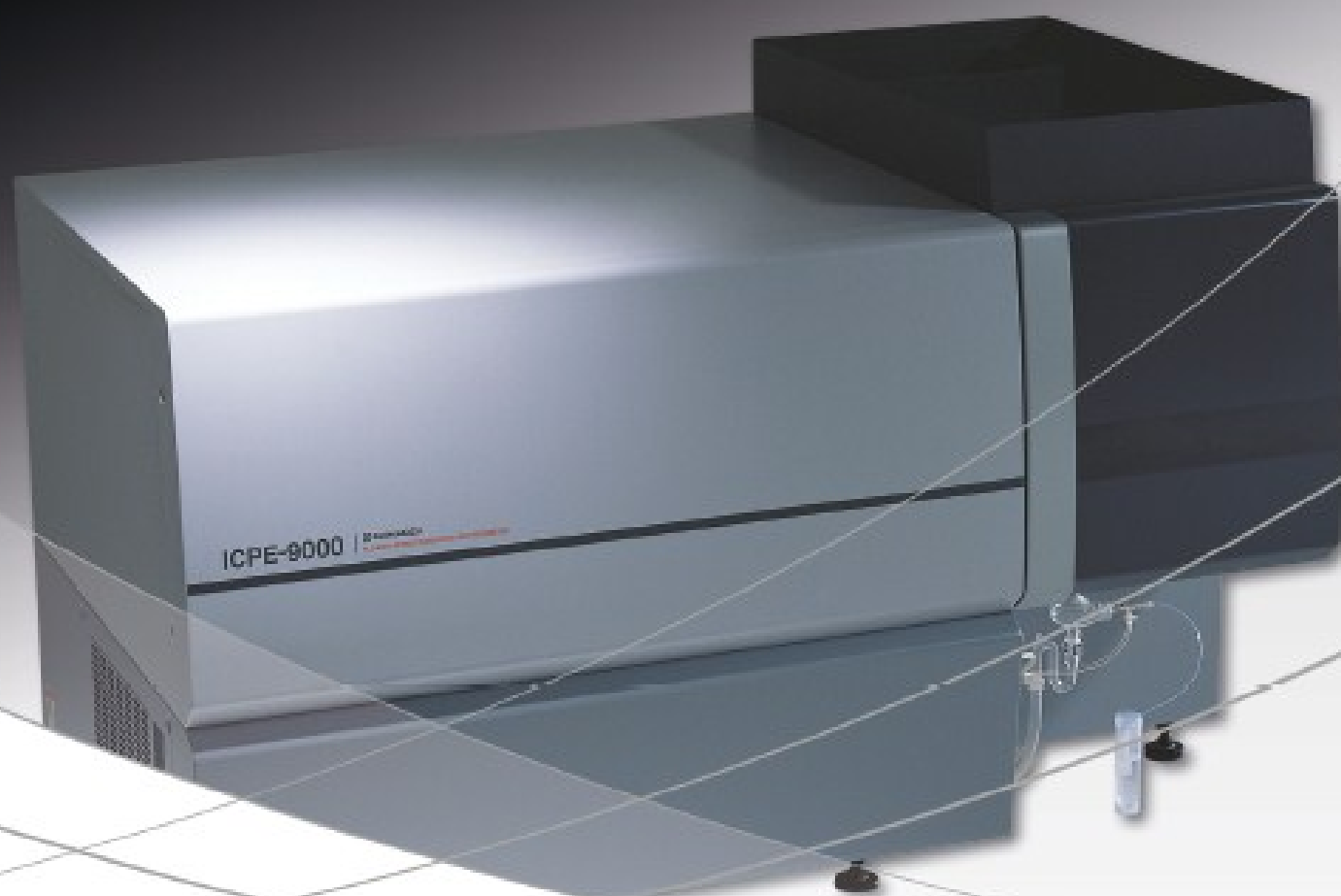
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