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   - Session III: Isotope ratio measurements ..................................................................... 11
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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 2016 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 boarders 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
### 3 Organising and Scientific Committee

- **Yngvar Thomassen** (Chairman), National Institute of Occupational Health, Oslo and Norwegian University of Life Sciences, Ås, Norway
- **Elin Gjengedal**, Norwegian University of Life Sciences, Ås, Norway
- **Balazs Berlinger**, National Institute of Occupational Health, Oslo, Norway
- **Ivar Martinsen**, GE Healthcare, Oslo, Norway
- **Arne Åsheim** (Exhibition Coordinator), Molab AS, Department Porsgrunn, Norway
- **Sverre Omang**, Oslo, Norway
- **Oddvar Røyseth** (Treasurer), Røyseth AS, 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:

<table>
<thead>
<tr>
<th>Day</th>
<th>Date</th>
<th>Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Saturday</td>
<td>June 4</td>
<td>16:00 - 18:00</td>
</tr>
<tr>
<td>Sunday</td>
<td>June 5</td>
<td>07:30 - 08:15</td>
</tr>
<tr>
<td>Monday</td>
<td>June 6</td>
<td>07:30 - 08:00</td>
</tr>
<tr>
<td>Tuesday</td>
<td>June 7</td>
<td>07:30 - 08:00</td>
</tr>
<tr>
<td>Wednesday</td>
<td>June 8</td>
<td>08:30 - 09:00</td>
</tr>
</tbody>
</table>

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

Homepage: 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, June 4, 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 and PerkinElmer.

Sunday, June 5, 14:00: 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 600 (not included in the Registration Fee).

Sunday, June 5, 14:00: Guided walk to Bødal Glacier

On the Bødal 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 Bødal Valley, with the Bødalseter summer pasture farms and the Bødal Glacier, is one of the gems of the Jostedalsbreen 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 5, 14:00: 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 6, 21:30: Bring your own wine

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

Tuesday, June 7, 16:15: Excursion to the Briksdal Glacier with the 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 aquavit 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. Included in the registration fee.

This event is kindly sponsored by Thermo Fisher Scientific, NerliensMeszansky, Nu Instruments and Shimadzu.
6 Scientific Programme

Oral presentations
Invited plenary lectures and submitted oral contributions will be 25 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 5, 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)*, *Journal of Analytical Atomic Spectrometry (JAAS)* and the Analytical Section of the Norwegian Chemical Society.

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:

- Elemental Scientific Inc.
- Holger Hartmann AS/ PerkinElmer
- Agilent Technologies/Matriks AS
- Nu Instruments
- Shimadzu Europa GmbH
- Thermo Fisher Scientific /NerliensMeszansky AGA

9 Correspondence after the Conference

Yngvar Thomassen
National Institute of Occupational Health
P.O. Box 8149 DEP, N-0033 Oslo, Norway
Tel: +47 23 10 53 20  Fax: +47 23 19 52 06
E-mail: Yngvar.Thomassen@stami.no
# Schedule of Events

## Saturday, June 4, 2016

<table>
<thead>
<tr>
<th>Time</th>
<th>Event</th>
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</thead>
<tbody>
<tr>
<td>16:00 - 18:00</td>
<td>Registration</td>
</tr>
</tbody>
</table>
| 17:00 - 19:00 | **Informal get-together in the Hotel Alexandra Bath & Spa**  
This event is kindly sponsored by Holger Hartmann AS and PerkinElmer |
| 19:00     | Dinner                                                              |

## Sunday, June 5, 2016

<table>
<thead>
<tr>
<th>Time</th>
<th>Event</th>
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<tbody>
<tr>
<td>07:30 - 08:15</td>
<td>Registration</td>
</tr>
<tr>
<td>08:15 - 08:30</td>
<td>Welcome/Opening remarks</td>
</tr>
</tbody>
</table>
| 08:30 - 12:55 | Plenary Session I: Progress in plasma spectrochemistry  
Poster viewing/exhibition                                           |
| 13:00        | Lunch                                                                |
| 14:00        | Excursion to Geiranger/The Skåla Challenge/Guided walk to Bødal Glacier |
| 19:00        | Dinner                                                              |

## Monday, June 6, 2016

<table>
<thead>
<tr>
<th>Time</th>
<th>Event</th>
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| 08:00 - 10:05 | Plenary Session II: Chemical imaging analysis  
Poster viewing/exhibition |
| 10:25 - 11:45 | Plenary Session III: Isotope ratio measurements  
Poster viewing/exhibition |
| 11:45 – 13:00 | Plenary Session IV: Applications of plasma spectrometry               |
| 13:00 - 14:15 | Lunch                                                                |
| 14:15 – 15:30 | Plenary Session V: Progress in glow discharge and laser ablation mass spectrometry  
Poster viewing/exhibition |
## Schedule of Events

### Short Course Session 1

<table>
<thead>
<tr>
<th>Time</th>
<th>Course A1</th>
<th>Course A2</th>
<th>Course A3</th>
<th>Course A4</th>
<th>Course A5</th>
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<tbody>
<tr>
<td>15:45 - 17:30</td>
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### Short Course Session 2

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<tr>
<th>Time</th>
<th>Course B1</th>
<th>Course B2</th>
<th>Course B3</th>
<th>Course B4</th>
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<tbody>
<tr>
<td>17:45 - 19:30</td>
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<tr>
<td>19:45</td>
<td>Dinner</td>
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</tr>
<tr>
<td>21:00</td>
<td>Poster viewing and discussions with wine tasting (Bring your own wine)</td>
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</table>

This event is sponsored by Agilent Technologies and Matriks AS.

#### Tuesday, June 7, 2016

<table>
<thead>
<tr>
<th>Time</th>
<th>Event</th>
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<tbody>
<tr>
<td>08:00 - 08:15</td>
<td>The Nordic Plasma Torch Award</td>
</tr>
<tr>
<td>08:15 – 11:40</td>
<td>Session VI: Speciation analysis and applications of plasma spectrochemistry Poster viewing/exhibition</td>
</tr>
<tr>
<td>11:40 – 12:45</td>
<td>Session VII: New analytical capabilities Poster viewing/exhibition</td>
</tr>
<tr>
<td>13:00 - 14:10</td>
<td>Lunch</td>
</tr>
</tbody>
</table>

### Short Course Session 3

<table>
<thead>
<tr>
<th>Time</th>
<th>Course C1</th>
<th>Course C2</th>
<th>Course C3</th>
<th>Course C4</th>
</tr>
</thead>
<tbody>
<tr>
<td>14:10 - 15:55</td>
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<tr>
<td>16:15</td>
<td>Excursion to Briksdal Glacier/Conference Outdoor Dinner</td>
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</tbody>
</table>

These events are sponsored by Thermo Fischer Scientific, NerliensMeszansky, Nu Instruments and Shimadzu.

#### Wednesday, June 8, 2016

### Short Course Session 4

<table>
<thead>
<tr>
<th>Time</th>
<th>Course D1</th>
<th>Course D2</th>
<th>Course D3</th>
<th>Course D4</th>
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<tbody>
<tr>
<td>09:15 - 11:00</td>
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<tr>
<td>11:15 – 11:30</td>
<td>Closing remarks and farewell</td>
<td></td>
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<tr>
<td>12:00 -</td>
<td>Lunch</td>
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</table>
**Daily Programme**

**Sunday, June 5, 2016**

<table>
<thead>
<tr>
<th>Time</th>
<th>Abstr.</th>
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</table>
| 08:15-08:30 | Welcome/Opening remarks  
Yngvar Thomassen, Conference chairman |
| 08:30-09:00 | Distinguished Speaker's Lecture: Inductively coupled plasma: From analytical applications to additive manufacturing  
| 09:00-09:25 | Is there no better plasma for spectrochemistry than the argon plasma?  
Gary M. Hieftje, Andrew J. Schwartz, Yan Cheung, Cera E. Cissna, and Gina C. Roesch, Department of Chemistry, Indiana University, Bloomington, USA |
| 09:25-09:50 | Dielectric barrier discharge – From soft ionization to element excitation  
Sebastian Brandt, Alexander Schütz, David Klute, Antje Michels, Vlasta Horvatic, Ced Vadal and Joachim Franzke  
1 University of Zagreb, Croatia, 2 ISAS - Leibniz-Institut für Analytische Wissenschaften, Dortmund, Germany |
| 09:50-10:15 | Hot and cold plasma for direct analysis of single nanoparticles, liquid crystal displays, and more  
Carsten Engelhard, University of Siegen, Department of Chemistry and Biology, Germany |
| 10:15-10:45 | Coffee, exhibition and poster viewing |
| 10:45-11:10 | New developments in the analysis of composite nanoparticles using single particle ICP-MS and field-flow-fractionation  
James F. Ranville and Angela Barber, Department of Chemistry, Colorado School of Mines, USA |
| 11:10-11:25 | Analysis of hydrogen, carbon and oxygen by GD-OES  
Volker Hoffmann, IFW Dresden, Institute for Complex Materials, Germany |
| 11:25-11:50 | Distance of flight mass spectrometry and new modes of time-of-flight mass spectrometry: Developing new analytical capabilities through novel instrumental approaches  
Steven Ray, Elise Dennis, Gary M. Hieftje, Christie Enke and David Koppenaal  
1 Department of Chemistry, State University of New York at Buffalo, USA  
2 Department of Chemistry, Indiana University, Bloomington, USA  
3 Department of Chemistry, New Mexico State University, Albuquerque, NM  
4 Pacific Northwest National Laboratory, Richland, WA, USA |
| 11:50-12:15 | Plasma-based mass spectrometry to characterize ultrafine particle exposure and reactivity in occupational environments  
Davy Rousset, National Institute of Occupational Health & Safety (INRS), Vandoeuvre cedex, France |
Sunday, June 5, 2016 cont.

<table>
<thead>
<tr>
<th>Time</th>
<th>Abstr.</th>
<th>Title</th>
<th>Authors</th>
</tr>
</thead>
<tbody>
<tr>
<td>12:15-12:40</td>
<td>O-9</td>
<td>Comparison of load coil and flat plasmas</td>
<td>Ari Väisänen, Siiri Perämäki and Roshan Budhathoki, Department of Chemistry, University of Jyväskylä, Finland</td>
</tr>
</tbody>
</table>
| 12:40-12:55 | O-10  | Diagnostics and simulation of a low-argon-flow ICP followed by optimization of the torch | M. Voronov$^1$, V. Hoffmann$^2$, C. Engelhard$^3$ and W. Buscher$^1$  
$^1$ University of Münster, Institute of Inorganic and Analytical Chemistry, Germany  
$^2$ IFW Dresden, Institute for Complex Materials, Dresden, Germany  
$^3$ University of Siegen, Department of Chemistry & Biology, Germany |
| 13:00 |       | Lunch                                                                 |                                                                            |
| 14:00-19:00 |       | Excursion to Geiranger, the Skåla challenge and guided walk to Bødal Glacier |                                                                            |
| 19:00 |       | Dinner                                                                |                                                                            |

Monday, June 6, 2016

Session II: Chemical imaging analysis

Chairman: Freddy Adams

<table>
<thead>
<tr>
<th>Time</th>
<th>Abstr.</th>
<th>Title</th>
<th>Authors</th>
</tr>
</thead>
<tbody>
<tr>
<td>08:00-08:25</td>
<td>O-11</td>
<td>Recent advances in mass cytometry and tissue imaging</td>
<td>Dmitry R. Bandura, Alexander Loboda, Vladimir I. Baranov, Olga I. Ornatsky, Adam Carew, Qing Chang, Tauvia Closson, Daaf Sandkuijl, Eric Swanson and Andrei Terekid, Fluidigm Canada Inc., Markham, Ontario, Canada</td>
</tr>
<tr>
<td>08:25-08:50</td>
<td>O-12</td>
<td>Multi-element bioimaging of plant tissues</td>
<td>Daniel Pergament Persson, Anle Chen, Jan K. Schjoerring and Søren Husted, Department of Plant and Environmental Sciences (PLEN), Faculty of Science, University of Copenhagen, Denmark</td>
</tr>
</tbody>
</table>
| 08:50-09:05 | O-13  | Laser ablation-optical emission spectrometry for elemental bioimaging | Michael Holtkamp$^1$, Georgina M. Thyssen$^1$, Mandy Großgarten$^1$, Dörthe Dietrich$^1$, Michael Sperling$^1$ and Uwe Karst$^1$  
$^1$ University of Münster, Institute of Inorganic and Analytical Chemistry, Germany  
$^2$ EVISA, European Virtual Institute for Speciation Analysis, Münster, Germany |
| 09:05-09:20 | O-14  | Imaging of nanoparticles in cells by LA-ICP-MS                       | Heike Traub$^1$, Daniela Drescher$^{1,2}$, Tina Büchner$^2$, Ingrid Zeise$^2$, Janina Kneipp$^{1,2}$ and Norbert Jakubowski$^1$  
$^1$ Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin, Germany  
$^2$ Humboldt-Universität zu Berlin, Department of Chemistry, Germany |
| 09:20-09:35 | O-15  | Single cell ICP-MS quantification of metal content in individual cells - An insight into cancer treatment | Chady Stephan and Erik Buseth, PerkinElmer, Shelton, CT, USA  
and Lauren Amable, National Institute on Minority Health and Health Disparities (NIMHD), Bethesda, USA |
### Monday, June 6, 2016 cont.

<table>
<thead>
<tr>
<th>Time</th>
<th>Abstr.</th>
<th>Title</th>
<th>Authors</th>
</tr>
</thead>
</table>
| 09:35-09:50| 0-16   | Quantitative bioimaging by means of LA-ICP-MS reveals silver migration from silver-coated endoprostheses into the patient's brain | Mandy Großgarten\(^1\), Ann-Christin Niehoff\(^2\), Astrid Jeibmann\(^2\), Michael Sperling\(^3\), Werner Paulus\(^4\) and Uwe Karst\(^3\)  
\(^1\) University of Münster, Institute of Inorganic and Analytical Chemistry, Germany  
\(^2\) University Hospital Münster, Institute of Neuropathology, Münster, Germany  
\(^3\) EVISA, European Virtual Institute for Speciation Analysis, Münster, Germany |
| 09:50-10:05| 0-17   | Quantitative bioimaging of platinum via online isotopic dilution-LA-ICP-MS | Oliver Bolle Bauer\(^1\), Michael Sperling\(^1\), Hans-Joachim Schurek\(^2\), Giuliano Ciarimboli\(^2\), Uwe Karst\(^1\)  
\(^1\) Institute of Inorganic and Analytical Chemistry, University of Münster, Germany  
\(^2\) Department of Experimental Nephrology, University Hospital of Münster, Germany |
| 10:05-10:25|        | Coffee break, exhibition and poster viewing                         |                                                                        |

#### Session III: Isotope ratio measurements
Chairman: Klaus Heumann

<table>
<thead>
<tr>
<th>Time</th>
<th>Abstr.</th>
<th>Title</th>
<th>Authors</th>
</tr>
</thead>
<tbody>
<tr>
<td>10:25-10:50</td>
<td>0-18</td>
<td>Measurements of isotope abundances in laser plasmas</td>
<td>Richard E. Russo, Lawrence Berkeley National Laboratory, Berkeley, USA</td>
</tr>
</tbody>
</table>
| 10:50-11:15| 0-19   | Variability in trace element isotope composition in environmental matrices | Ilia Rodushkin\(^1,2\), Nicola Pallavicini\(^1,2\), Emma Engström\(^1,2\) and Douglas C. Baxter\(^2\)  
\(^1\) Division of Geosciences, Luleå University of Technology, Luleå, Sweden  
\(^2\) ALS Laboratory Group, ALS Scandinavia AB, Aurorum 10, Luleå, Sweden |
| 11:15-11:30| 0-20   | Determination of long-lived radionuclides at sub ng.kg\(^-1\) levels in presence of high amount of isobaric interferences by ICP-QQQ-MS | Panayot Petrov\(^1\), Ben Russell\(^2\), David Douglas\(^1\), Patrick (Paddy) Rega\(^3\) and Heidi Goenaga-Infante\(^1\)  
\(^1\) LGC, Teddington, Middlesex  
\(^2\) NPL, Teddington, Middlesex  
\(^3\) University of Surrey, Guildford, Surrey, UK |
| 11:30-11:45| 0-21   | Isotopic fractionation of natural uranium in a human neuronal cell model: A proof of concept | Eduardo Paredes, Emilie Avazeri, Véronique Malard, Claude Vidaud, Pascal Reiller, Richard Ortega, Anthony Nonell, Helene Isnard, Frederic Chartier and Carole Bresson, CEA Saclay, DEN/DANS/DPC/SEARS/LANIE, Laboratoire de développement Analytique Nucléaire Isotopique et Élémentaire, Gif sur Yvette, France |

#### Session IV: Applications of plasma spectrometry
Chairman: Carsten Engelhard

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<thead>
<tr>
<th>Time</th>
<th>Abstr.</th>
<th>Title</th>
<th>Authors</th>
</tr>
</thead>
</table>
| 11:45-12:00| 0-22   | Determination of iron, nickel and vanadium in petroleum heavy oil and residue samples by MP-AES | Jenny Nelson\(^1\), Greg Gilleland\(^1\), Steve Wall\(^1\), Laura Poirier\(^2\), Lidia Berhane\(^2\) and Francisco Lopez-Linares\(^2\)  
\(^1\) Agilent Technologies, Inc., Santa Clara, CA, USA  
\(^2\) Chevron Energy Technology Company, Richmond, CA, USA |
Monday, June 6, 2016 cont.

Time | Abstr. | Title | Authors | Institutions
--- | --- | --- | --- | ---
12:00-12:15 | O-23 | Rare earth metals in biomass fly ash – Analysis using ICP-OES | S. Perämäki and A. Väisänen, Department of Chemistry, University of Jyväskylä, Finland

12:15-12:30 | O-24 | Trans-boundary atmospheric transport of toxic metals to Norway 1976-2016 | Eiliv Steinnes, Department of Chemistry, Norwegian University of Science and Technology, Trondheim, Norway

12:30-12:45 | O-25 | Quantitative determination of major-minor-and trace elements in beer using ICP-OES spectrometry | Uwe Oppermann\(^1\), Jan Knoop\(^1\), Johan Leinders\(^1\) and Jürgen Schram\(^2\)
\(^1\)Shimadzu Europa GmbH, Duisburg, Germany
\(^2\)University of Applied Sciences, Krefeld, Germany

12:45-13:00 | O-26 | Sequential extraction of potentially toxic elements in sediments and speciation of arsenic in sediments and water | Abayneh Ataro Ambushe\(^1\), Mokgehle Refiloe Letsoalo\(^1\), Dithobolong Lovia Matabane\(^1\) and Taddese Wondimu Godeto\(^2\)
\(^1\)Department of Chemistry, University of Limpopo, Sovenga, South Africa
\(^2\)Department of Chemistry, University of Johannesburg, Johannesburg, South Africa

13:00-14:00 | | Lunch |

Session V: Progress in glow discharge and laser ablation mass spectrometry
Chairman: Steven Ray

Time | Abstr. | Title | Authors | Institutions
--- | --- | --- | --- | ---
14:15-14:40 | O-27 | State of the art and perspectives of plasma-based mass spectrometry techniques for direct solid analysis at the micro and nanoscale | Rosario Pereiro, Beatriz Fernández, Lara Lobo, Maria Cruz-Alonso, Sara M. Rodriguez, Rocío Muñiz and Alfredo Sanz-Medel, Department of Physical and Analytical Chemistry, University of Oviedo, Spain

14:40-15:05 | O-28 | Analysis of hard-to-digest ceramics by using laser ablation in liquid | Naoki Furuta, Chuo University, Department of Applied Chemistry, Tokyo, Japan

15:05-15:30 | O-29 | ICP-TOF-MS for comprehensive elemental analysis of discrete samples: Application in laser ablation and single-nanoparticle analysis | Alexander Gundlach-Graham\(^1\), Lyndsey Hendriks\(^1\), Benita Ramkorun-Schmidt\(^2\), Marcel Burger\(^1\), Gunnar Schwarz\(^1\), Antonia Praetorius\(^3\), Eli Goldberg\(^1\), Frank von der Kammer\(^3\), Bodo Hattendorf\(^1\) and Detlef Günther\(^1\)
\(^1\)Department of Chemistry and Applied Biosciences, ETH Zurich, Switzerland
\(^2\)BAM Federal Institute for Materials Research and Testing, Berlin, Germany
\(^3\)Department of Environmental Geosciences, University of Vienna, Austria

15:15-15:30 | O-30 | A time resolved study of thin coated and multi-layered composite materials using Astrum GDMS | DeAnn Barnhart\(^1\), Glyn Churchill\(^1\), Andrew Burrows\(^1\) and Bengt Sandgärde\(^2\)
\(^1\)Nu Instruments Limited, Wrexham, Clwyd, UK
\(^2\)Massanalyt Spectrometry Nordic AB, Farsta, Sweden
### Monday, June 6, 2016 cont.

#### Short Course Session 1

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<tr>
<td>17:30-</td>
<td>Richard Russo:</td>
<td>Laser ablation ICP-MS with LIBS and LAMIS</td>
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<td>17:45-</td>
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- Coffee break, exhibition and poster viewing

#### Short Course Session 2

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<td>19:00</td>
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<td>19:45</td>
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- Dinner

- Poster viewing and discussions with wine tasting
  - Bring your own wine
  - This event is sponsored by Agilent Technologies and Matriks AS
**Tuesday, June 7, 2016**

<table>
<thead>
<tr>
<th>Time</th>
<th>Abstr.</th>
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<tbody>
<tr>
<td>08:00-08:15</td>
<td>The Nordic Plasma Torch Award</td>
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<td></td>
<td>Organising Committee</td>
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<tr>
<td></td>
<td><strong>Session VI: Speciation analysis and applications of plasma spectrochemistry</strong></td>
</tr>
<tr>
<td></td>
<td>Chairman: Elin Gjengedal</td>
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<tr>
<td>08:15-08:40</td>
<td>Speciation analysis with ICP-MS detection in drug development</td>
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<tr>
<td></td>
<td>Bente Gammelgaard, Department of Pharmacy, University of Copenhagen,</td>
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<td>Denmark</td>
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<tr>
<td>08:40-09:05</td>
<td>ICP-MS based techniques to track for uranium molecular targets in</td>
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<td></td>
<td>reproductive organs of Zebrafish</td>
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<td></td>
<td>Sandra Mounicou(^1), Yvan Eb-Levadoux(^2), Carine Arnauguilhem(^1),</td>
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<td></td>
<td>Olivier Simon(^2), Ryszard Lobinski(^1) and Sandrine Frelon(^2).</td>
</tr>
<tr>
<td></td>
<td>(^1) Laboratoire de Chimie Analytique Bio-Inorganique et Environnement,</td>
</tr>
<tr>
<td></td>
<td>Pau, France</td>
</tr>
<tr>
<td></td>
<td>(^2) Laboratoire de Biogéochimie, Biodisponibilité et Transfert des</td>
</tr>
<tr>
<td></td>
<td>radionucléides, St Paul lez Durance Cedex, France</td>
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<tr>
<td>09:05-09:30</td>
<td>Speciation analysis and element-bioimaging: From genetic diseases to</td>
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<td></td>
<td>new implants</td>
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<td></td>
<td>Michael Sperling, D. Oliver Hachmoller, Stefanie Fingerhut and Uwe</td>
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<td></td>
<td>Karst, Institut für Anorganische und Analytische Chemie, University of</td>
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<tr>
<td></td>
<td>Münster, Germany</td>
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<tr>
<td>09:30-09:55</td>
<td>Speciation of organophosphates and fluorophosphates in lithium ion</td>
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<td>battery electrolytes</td>
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<td></td>
<td>Sascha Nowak, Vadim Kraft, Yannick Stenzel, Jennifer Menzel and Martin</td>
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<td></td>
<td>Winter, MEET Battery Research Center, University of Münster, Germany</td>
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<tr>
<td>09:55-10:20</td>
<td>A novel approach for speciation of Cr(^{3+}) and Cr(^{6+}) in total</td>
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<td>particulate matter from mainstream tobacco smoke utilizing ETV-ICP-MS</td>
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<td></td>
<td>R. Steven Pappas(^1) Mark R. Fresquez(^2) and Clifford H. Watson(^1),</td>
</tr>
<tr>
<td></td>
<td>(^1) Centers for Disease Control &amp; Prevention, Tobacco and Volatiles</td>
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<tr>
<td></td>
<td>Branch, Atlanta, USA, (^2) Battelle – Atlanta Analytical Services,</td>
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<tr>
<td></td>
<td>Atlanta, USA</td>
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<tr>
<td>10:20-10:40</td>
<td>Coffee break, exhibition and poster viewing</td>
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<tr>
<td>10:40-10:55</td>
<td>The EU 2015/1006 regulation and its impact on arsenic foodstuff</td>
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<td></td>
<td>speciation and quantification</td>
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<td></td>
<td>Simon Nelms, Thermo Fisher Scientific, Hemel Hempstead, UK</td>
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<tr>
<td>10:55-11:10</td>
<td>Use of seaweed in food and feed – implications for food/feed safety</td>
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<tr>
<td></td>
<td>Jens J Sloth, Susan L. Holdt and Rie R. Rasmussen, National Food Institute,</td>
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<td></td>
<td>Technical University of Denmark, Søborg, Denmark</td>
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<tr>
<td>11:10-11:25</td>
<td>Detection of metal-based nanoparticles in environmental and biological</td>
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<td>matrices using single particle ICP-MS</td>
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<td>Chady Stephan and Erik Buseth, PerkinElmer, Shelton, CT, USA</td>
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<td>11:25-11:40</td>
<td>Influence of rapeseed cake supplement in cow feed on iodine in milk</td>
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<td>Anicke Brandt-Kielsen, Øyvind Enger, Gisken Træn, Egil Prestløkken and</td>
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<td>Brit Salbu, Norwegian University of Life Sciences, Ås, Norway</td>
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</tbody>
</table>
Tuesday, June 7, 2016 cont.

**Session VII: New analytical capabilities**

Chairman: Gary Hieftje

<table>
<thead>
<tr>
<th>Time</th>
<th>Abstr.</th>
<th>Title</th>
<th>Authors</th>
</tr>
</thead>
<tbody>
<tr>
<td>11:40-</td>
<td>O-40</td>
<td>Improving ICP accuracy for the analysis of bioethanol samples by applying a new total consumption sample introduction device</td>
<td>José Luis Todolí¹, Carlos Sánchez² and Charles-Philippe Lienemann²</td>
</tr>
<tr>
<td>12:05</td>
<td></td>
<td></td>
<td>¹Department of Analytical Chemistry, Nutrition and Food Sciences, Alicante, Spain. ²IFP Energies Nouvelles, Solaize, France</td>
</tr>
<tr>
<td>12:05-</td>
<td>O-41</td>
<td>High-resolution chemical profiling of heterogeneous materials using laser ablation ionization mass spectrometry (LIMS)</td>
<td>A. Riedo¹,² V. Grimaudo³, P. Moreno-García³, M.B. Neuland², M. Tulej², P. Broekmann³ and P. Wurz²</td>
</tr>
<tr>
<td>12:30</td>
<td></td>
<td></td>
<td>¹Leiden Observatory, University of Leiden, The Netherlands ²Space Research and Planetary Sciences, University of Bern, Switzerland ³Department of Chemistry and Biochemistry, University of Bern, Switzerland</td>
</tr>
<tr>
<td>12:30-</td>
<td>O-42</td>
<td>Development of triple quadrupole ICP-MS performance for difficult applications</td>
<td>Uwe Noetzel¹, Naoki Sugiyama² and Glenn Woods³</td>
</tr>
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<td>12:45</td>
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<td>¹Agilent Technologies Germany; ²Agilent Technologies Japan; ³Agilent Technologies (UK) Ltd</td>
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<td>13:00-</td>
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<td>Lunch</td>
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<td>13:00-14:10</td>
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**Short Course Session 3**

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<th>C3:</th>
<th>C4:</th>
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<tbody>
<tr>
<td>14:10-15:55</td>
<td>C1- C4</td>
<td>Carsten Engelhard: Plasma-based sources for direct analysis: Fundamentals and applications of ambient desorption/ionization mass spectrometry</td>
<td>Rosario Pereiro: Fundamentals and applications of depth profile analysis by glow discharge spectrometries for beginners</td>
<td>Davy Rousset: How to collect relevant ambient and occupational air samples for plasma based spectrochemical analysis</td>
<td>Ilia Rodushkin: Sources of contamination and remedial strategies</td>
</tr>
</tbody>
</table>

**Excursion to Briksdal Glacier and conference outdoor dinner**

This event is sponsored by Thermo Fischer Scientific, NerliensMeszansky, Nu Instruments and Shimadzu
### Wednesday, June 8, 2016

#### Short Course Session 4

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<tr>
<th>Time</th>
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<th>D3:</th>
<th>D4:</th>
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<tbody>
<tr>
<td>09:15-11:00</td>
<td>D1- D4</td>
<td>Dmitry Bandura:</td>
<td>Sandra Mounicou:</td>
<td>José-Luis Todoli:</td>
<td>A.Gundlach-Graham and S. Ray:</td>
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<td></td>
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<td>Introduction to mass cytometry</td>
<td>Analytical strategies for metalloproteins analysis</td>
<td>Matrix effects in ICP techniques</td>
<td>Part 2: Plasma-source time-of-flight and distance-of-flight mass spectrometry</td>
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<td>11:00-11:15</td>
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<td>Coffee break</td>
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<tr>
<td>11:15-11:30</td>
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<td>Closing remarks and farewell</td>
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<td>12:00-</td>
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<td>Lunch</td>
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</table>
12 Poster Presentations

Abstr.    Sunday, June 5 - Wednesday, June 8, 2016

P-1  A METROLOGY STRATEGY FOR THE ACCURATE DETERMINATION OF CHROMIUM (VI) IN WATER USING HPLC-ICP-MS WITH SPECIES SPECIFIC ISOTOPE DILUTION ANALYSIS. 
John Entwisle, Sarah Hill, Panayot Petrov and Heidi Goenaga-Infante 
LGC Ltd, Middlesex, UK

P-2  DETERMINATION OF METHYLMERCURY AND INORGANIC MERCURY SPECIES IN SEAFOOD BY HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC) COUPLED TO AN ICP-MS 
E. A. Nebera, A. A. Komarov 
Russian State Center for Quality and Standardization of Veterinary Drugs and Feeds, Moscow, Russia

P-3  DETERMINATION OF PALLADIUM IN ENVIRONMENTAL SAMPLES BY ICP-MS TECHNIQUES 
Terhi Suoranta1, Syed Nadeem Hussain Bokhari2, Thomas Meisel1, Matti Niemelä1 and Paavo Perämäki1 
1 Research Unit of Sustainable Chemistry, University of Oulu, Finland 
2 General and Analytical Chemistry, Montanuniversität Leoben, Austria

P-4  COMPARISON OF THE INFLUENCES OF DIFFERENT LITHIUM ION BATTERY ADDITIVES ON THE ELECTROLYTE AGING BY SIMULTANEOUS ONLINE COUPLING OF TWO DIMENSIONAL ION CHROMATOGRAPHY TO INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY AND ELECTROSpray IONIZATION MASS SPECTROMETRY 
Jennifer Menzel, Vadim Kraft, Martin Winter and Sascha Nowak, University of Münster, MEET Battery Research Center, Germany

P-5  DEVELOPMENT OF ANALYTICAL STRATEGIES TO STUDY SELENOPROTEIN EXPRESSION IN HUMAN CELL LINES 
Jordan Sonet, Katarzyna Bierla, Sandra Mounicou, Ryszard Lobinski and, Anne-Laure Bulteau and Laurent Chavatte 
CNRS/UPPA, Laboratoire de Chimie Analytique Bio-inorganique et Environnement, Pau, France

P-6  SPATIALLY RESOLVED ANALYSIS OF THE LITHIUM DISTRIBUTION ON CYCLED ELECTRODES BY LASER ABLATION – INDUCTIVELY COUPLED PLASMA - OPTICAL EMISSION SPECTROMETRY (LA-ICP-OES) 
Constantin Lürenbaum, Britta Vortmann, Martin Winter and Sascha Nowak 
University of Münster, MEET Battery Research Center, Germany

P-7  ADAPTION OF GLOW-DISCHARGE SECTOR FIELD MASS SPECTROMETRY IN THE FIELD OF BATTERY RESEARCH 
Marco Evertz, Timo Schwieters, Markus Börner, Martin Winter and Sascha Nowak 
University of Münster, MEET Battery Research Center, Germany

P-8  SYNTHESIS AND APPLICATION OF A MATRIX MATCHED EXTERNAL STANDARD MATERIAL TO QUANTIFY MANGANESE IN LITHIUM ION BATTERY ELECTRODES BY MEANS OF LASER ABLATION INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY 
Timo Schwieters, Marco Evertz, Martin Winter and Sascha Nowak 
University of Münster, MEET Battery Research Center, Germany

P-9  INVESTIGATION OF LITHIUM-ION BATTERY ELECTROLYTES BY A GAS CHROMATOGRAPHY - BARRIER IONIZATION DISCHARGE DETECTOR 
X. Mönnighoff, C. Lürenbaum, M. Winter and S. Nowak 
University of Münster, MEET Battery Research Center, Germany
ABSTRACTS

**P-10** QUANTIFICATION OF ORGANOPHOSPHATES AS AGING PRODUCTS OF LITHIUM-ION BATTERY ELECTROLYTES BY MEANS OF GAS CHROMATOGRAPHY - INDUCTIVELY COUPLED PLASMA SECTOR FIELD MASS SPECTROMETRY (GC-ICP-SF-MS)
Y. P. Stenzel, C. Lürenbaum, V. Kraft, M. Winter and S. Nowak
University of Münster, MEET Battery Research Center, Germany

**P-11** NANO-PARTICLE ANALYSIS USING DATA ACQUISITION DWELL TIMES BETWEEN 10µS AND 50µS WITH A DYNAMIC RANGE EQUIVALENT TO MORE THAN 1E9 CPS
Phil Shaw¹, Ariane Donard¹ and Bengt Sandgarde²
¹ Nu Instruments, Wrexham Industrial Estate, Wrexham, UK
² MS Nordic, Farsta, Sweden

**P-12** 3D MODELLING OF PLASMA FLOW AND SPATIAL DISTRIBUTION OF TEMPERATURE IN THE PLASMACHEMICAL REACTOR
Kazan National Research Technical University named after A.N. Tupolev – KAI, Kazan, Russia

**P-13** 3D MODELLING AND OPTICAL DIAGNOSTICS OF GAS FLOW DYNAMICS NEAR THE ICP-MS INTERFACE
Kazan National Research Technical University named after A.N. Tupolev – KAI, Kazan, Russia

**P-14** STRATEGIES FOR DEALING WITH HIGH MATRIX SAMPLES USING ICP-MS
Marcus Manecki¹, Daniel Kutscher¹, Shona McSheehy Ducos¹ and Simon Nelms²
¹ Thermo Fisher Scientific, Bremen, Germany
² Thermo Fisher Scientific, Hemel Hempstead, UK

**P-15** EXPANDING THE DETECTABLE NANOPARTICLE SIZE RANGE BY SECTOR FIELD ICP-MS
Torsten Lindemann and Shona McSheehy Ducos
Thermo Fisher Scientific, Bremen, Germany

**P-16** USE OF 10¹³ OHM FARADAY CUP AMPLIFIERS IN (LA-) MC-ICP-MS: EXTERNAL PRECISION OF ²³⁴U/²³⁸U RATIOS AND Δ¹¹B
Nicholas S. Lloyd¹, Aleksey Yu. Sadekov² and Sambuddha Misra²
¹ Thermo Fisher Scientific, Bremen, Germany
² University of Cambridge, Department of Earth Sciences, Godwin Laboratory for Paleoclimate Research, UK

**P-17** INTERNAL STANDARDIZATION IN ICP-MS USING A DUAL MODE SAMPLE INTRODUCTION SYSTEM
Havia Johanna, Pietilä Heidi, Niemelä Matti and Perämäki Paavo
Research Unit of Sustainable Chemistry, University of Oulu, Finland

**P-18** THE DETERMINATION OF MERCURY IN PLASTICS BY COLD-VAPOR-GENERATION (CVG) INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETRY (ICP-OES).
Esa Lehtimäki* and Ari Väisänen
Department of Chemistry, University of Jyväskylä, Finland
Abstr.  Poster Presentations cont.

P-19  ADVANTAGES OF A HIGH TEMPERATURE TORCH INTEGRATED SAMPLE INTRODUCTION SYSTEM OVER A DESOLVATION SYSTEM FOR THE ANALYSIS OF MICROSAMPLES THROUGH ICP-MS
José-Luis Todolí,¹ Raquel Sánchez,¹ Águeda Cañabate,¹ Salvador Maestre,¹ Anthony Nonell,² Carole Bresson² et Frédéric Chartier³
¹Department of Analytical Chemistry, Nutrition and Food Science, University of Alicante, Spain
²CEA Saclay, DEN, DANS, DPC, SEARS, Laboratoire de développement Analytique Nucléaire Isotopique et Elémentaire, Gif-sur-Yvette, France
³CEA Saclay, DEN, DANS, DPC, Gif-sur-Yvette, France

P-20  LASER ABLATION COUPLED TO ICP TECHNIQUES FOR THE ANALYSIS OF CATALYSTS
José-Luis Todolí,¹ Ángela Villaseñor,¹ Caroline Greatti,² Marina Boccongelli²,
¹Department of Analytical Chemistry, Nutrition and Food Science, University of Alicante, Spain
²Total Research & Technology Gonfreville, Harfleur, France.

P-21  DETERMINATION OF RUBIDIUM IN DIFFICULT MATRICIES BY INDUCTIVE COUPLED PLASMA OPTICAL EMISSION SPECTROMETRY
E. Nygrén, S.Peramäki and A. Väisänen
University of Jyväskylä, Department of Chemistry, Finland

P-22  DEVELOPMENT OF AN LC-ICP-MS METHOD FOR ZINC SPECIATION IN FISH FEEDS
Marta Silva¹², Jens J. Sloth¹², Rune Waagbø¹², Robin Ørnsrud¹ and Heidi Amlund¹
¹National Institute of Nutrition and Seafood Research, Bergen, Norway
²Institute of Biology, University of Bergen, Bergen, Norway

P-23  DETERMINATION OF PHOSPHORUS, ALUMINUM AND IRON FROM SYNTHETIC MUNICIPAL WASTE WATER SLUDGE LEACHATE SAMPLES BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETRY ANALYSIS
Sini Reuna and Ari Väisänen
Department of Chemistry, University of Jyväskylä, Finland

P-24  ICP-OES FOR QUALITATIVE AND QUANTITATIVE ANALYSIS OF MINERAL ELEMENTS (P, K) IN THEIR RECOVERY FROM FLY ASH
Roshan Budhathoki and Ari Väisänen
Department of Chemistry, University of Jyväskylä, Finland

P-25  THERE IS A NEED FOR SPECIATION ANALYSIS OF SELENIUM IN FISH FEED AND FISH TISSUE
Veronika Sele¹, Jens J. Sloth², Robin Ørnsrud¹ and Heidi Amlund¹
¹National Institute of Nutrition and Seafood Research, Bergen, Norway
²National Food Institute, Technical University of Denmark, Søborg, Denmark

P-26  ELUCIDATION OF THE NEGATIVE MODE IONIZATION MECHANISM OF PERFLUORINATED COMPOUNDS USING AN ACTIVE CAPILLARY PLASMA IONIZATION SOURCE
Luzia Gyr and Renato Zenobi
Department of Chemistry and Applied Biosciences, ETH Zürich, Zürich, Switzerland.
Abstr.  Poster Presentations cont.

P-27  DEVELOPMENT OF A CHIP-BASED GRAPHENE-TITANIUM DIOXIDE NANOCOMPOSITE - ASSISTED PHOTOCATALYTIC REDUCTION DEVICE TO COUPLE HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY WITH INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY FOR ONLINE DETERMINATION OF INORGANIC SELENIUM SPECIES
Pei-Chun Chao,1 Tsung-Ting Shih2 and Yuh-Chang Sun1
1 Department of Biomedical Engineering and Environmental Sciences, National Tsing Hua University, Hsinchu, Taiwan
2 Material and Chemical Research Laboratories, Industrial Technology Research Institute, Hsinchu, Taiwan

P-28  ANALYSIS OF GAS TEMPERATURE AND OXYGEN EMISSION IN HIGH FREQUENCY ELECTRODELESS LIGHT SOURCES
Z. Gavare1,2, M.Zinge1, E. Bogans1 and A. Skudra1
1 Institute of Atomic Physics and Spectroscopy, University of Latvia, Riga, Latvia
2 Department of Physics, Faculty of Information Technologies, Latvia University of Agriculture, Jelgava, Latvia

P-29  ESTIMATION OF ELECTRON TEMPERATURE FROM EMISSION OF ARGON IN MULTI-ELEMENT CADMIUM AND ZINC LIGHT SOURCES
Z. Gavare1,2, M.Zinge1, E. Bogans1 and A. Skudra1
1 Institute of Atomic Physics and Spectroscopy, University of Latvia, Riga, Latvia
2 Department of Physics, Faculty of Information Technologies, Latvia University of Agriculture, Jelgava, Latvia

P-30  THE POTENTIAL OF DUAL PROBE ANALYSIS
Dick Waijers
Shell Technology Centre Amsterdam, Netherlands

P-31  MULTIELEMENTAL ANALYSIS OF PURIFIED AND TAB WATER SAMPLES WITH HR ICP-MS
T. Lavonen1 and E. Myllykylä1
1 VTT Oy Technical Research Centre of Finland, Espoo, Finland
INDUCTIVELY COUPLED PLASMA: FROM ANALYTICAL APPLICATIONS TO ADDITIVE MANUFACTURING


Kazan National Research Technical University named after A.N. Tupolev – KAI, Kazan, Russia
e-mail: albert.gilmudinov@kai.ru

Inductively coupled plasma (ICP) as a spectrally pure source of excited atoms and ions has been successfully used worldwide for the last thirty years in analytical emission and mass spectrometry, providing high performance, precision and record low detection limits. At the same time, ICP still has extensive technological potential of applications as a high purity material heating source on the one side and for the synthesis and modification of micro- and nano-powders with predefined properties, on the other.

In the last decade due to the rapid development of industrial additive laser technologies interest in plasma-chemical synthesis and processing of metallic and composite powders has greatly increased. With the development of advanced techniques such as supersonic laser cladding and 3-D printing the problem of plasma spheroidisation of powder materials is becoming extremely urgent task since these methods can only work properly with spherical particles. The range, fractional and chemical composition of powders for additive technologies are continuously expanding, what requires constant optimization of design and operating conditions of plasma-chemical reactors.

Empirically this optimization is extremely difficult to carry out, so the vast majority of researchers use advanced mathematical models of plasma-chemical systems with various degrees of complexity. Numerous experiments have shown that the plasma torch and its main parameters are characterized by a high degree of spatial and temporal heterogeneity. Therefore, three-dimensional and nonstationary model should be used to account for physical and chemical processes in the reactor and predict the properties of the final product.

The results of 3-D and nonstationary numerical modelling of the design and operating modes of industrial plasma-chemical reactors are given in the paper. Key features of industrial ICPs are compared to those of spectroanalytical plasma sources.

The work is supported by the Ministry of Education and Science of the Russian Federation (government contract №14.Z50.31.0023) and RFBR, project number 14-29-10281
IS THERE NO BETTER PLASMA FOR SPECTROCHEMISTRY THAN THE ARgon 
ICP?

Gary M. Hieftje, Andrew J. Schwartz, Yan Cheung, Cera E. Cissna, and Gina C. Roesch

Department of Chemistry, 800 East Kirkwood Ave., Bloomington IN 47405 USA
e-mail: Hieftje@indiana.edu

A clear trend in the search for alternatives to the conventional argon ICP is toward plasmas that have lower power and gas-flow requirements. The reasons are clear: more economical operation and possible on-site and field application. One option is to shrink the size of a discharge, but small size sometimes comes at a cost; sample atomization is often kinetically controlled and the kinetics are not strongly temperature-dependent. As a result, sensitivity can be lost and interferences become more troublesome. Another, perhaps more attractive option is to rely upon non-thermal atomization, by means of electrospraying or cathodic sputtering, both of which can be performed on very small platforms. Further, both of these atom-formation options can be employed in the open atmosphere, obviating the need for gas supplies or a flowing gas stream, and can operate at moderate power levels, commonly below 100W. A system will be described in this presentation that exploits these features.

A related move is toward relatively high-power plasmas for elemental analysis, but ones that are driven by low-cost microwave supplies and which can be operated in either nitrogen or air rather than argon. Performance levels are often somewhat disappointing, however, in comparison to those expected for the argon ICP, in part because of complex background spectra produced by the molecular support gas. Here, a novel power-coupling method will be introduced and its potential for both atomic emission and atomic mass spectrometry assessed.

Another important development is toward the use of plasmas for speciation or metallomic analysis. The most common approach is to couple the plasma to a prior separation method, such as liquid chromatography, electrophoresis, or ion-mobility spectrometry. However, such separation schemes can result in loss of metals from a protein, and can alter the speciation itself, by a change in equilibrium or in the local chemical environment of a metal during the separation process. A far surer approach is to employ a modulated or switched plasma source, with alternative operating modes, one mode providing elemental information and another the chemical form of the elements. In such operation, the speciation of each element is “frozen” at the moment of signal generation. Here, a novel mode of operation of a plasma source will be described that can provide elemental, speciation, or peptide-sequencing information upon command.
(O-3) DIELECTRIC BARRIER DISCHARGE – FROM SOFT IONIZATION TO ELEMENT EXCITATION

Sebastian Brandt, Alexander Schütz, David Klute, Antje Michels, Vlasta Horvatic*, Cedo Vadla* and Joachim Franzke

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Since 2007 ionization sources based on dielectric barrier discharges (DBD) intended for soft ionization of molecules have been and are still characterized. One of the methods that utilize these sources is the Dielectric Barrier Discharge Ionization (DBDI) and is based on a plasma jet established at the end of a capillary dielectric barrier discharge at atmospheric pressure. These discharges producing plasma jets that extend in the surrounding atmosphere are versatile, cheap and easy to construct devices that have found extensive use as ionization sources in the ambient mass spectrometry techniques.

A capillary He dielectric barrier discharge was investigated in respect to its performance as a soft or dissociative ionization source. Spatiotemporal measurements of the plasma emission showed that in one voltage duty cycle the plasma evolved from soft to dissociative ionization source. Earliest in time the soft plasma was generated between the electrodes as well as outside the capillary forming the plasma jet. It was characterized by significant radiation arising only from He and N$_2^+$, which are known to be important in the process of the soft ionization of the analyte. Later in time the plasma capable of dissociating molecules develops. It is characterized by appreciable radiation from analyte dissociation products, and is restricted to inter-electrode region in the capillary. Thus, for the soft ionization purposes, it is feasible to introduce the analyte exclusively in the plasma jet. For elemental analysis the inter-electrode plasma is appropriate.
HOT AND COLD PLASMAS FOR DIRECT ANALYSIS OF SINGLE NANOPARTICLES, LIQUID CRYSTAL DISPLAYS, AND MORE

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Are cold and hot plasmas really relevant to analytical chemistry? What is the best plasma source for my current analytical problem? This presentation aims to answer these two questions.

First, selected examples in plasma spectrochemistry are presented to highlight the applicability of low-temperature plasmas for ambient desorption/ionization molecular mass spectrometry (ADI-MS). In some cases, the sample should remain intact and minimal sample pre-treatment should be performed. Here, ADI-MS is considered especially attractive because only a few millimetre of the sample are typically probed and little to no sample preparation is required. ADI sources are useful in the characterization of lithium-ion-battery materials and can also be used to probe large-format liquid crystal displays. Recent results will be discussed.

Second, high-temperature plasmas such as the inductively coupled plasma (ICP) can be used to perform ultra trace elemental analysis. When coupled to a mass spectrometer and after careful optimization, ICPs can be used to detect single nanoparticles and to perform size characterization on a particle-by-particle basis. However, special attention has to be paid to the calibration, data acquisition, and data processing before accurate nanoparticle size distributions can be obtained. In many cases, results are biased by particle coincidence, split particle events, background signal, and varying concentrations. Here, our recent efforts to improve the overall performance of the ICP in nanoparticle detection and characterization will be discussed. A custom-built high-speed data acquisition unit specifically tailored to the needs of single-particle ICP-MS will be presented. It enables continuous acquisition of signals from discrete ion clouds in the ICP with microsecond time resolution and 100% duty cycle.
NEW DEVELOPMENTS IN THE ANALYSIS OF COMPOSITE NANOPARTICLES USING SINGLE PARTICLE ICP-MS AND FIELD-FLOW FRACTIONATION

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Release of engineered nanoparticles (ENPs) into the environment is inevitable as the nanotechnology industry continues to produce nanotechnology-enabled products (NEPs). Nanoparticles (NPs) are also becoming increasingly complex with some composite NPs containing multiple elements. In this work, advancements in single particle inductively coupled plasma (spICP-MS), used in tandem with the particle separation capabilities of field-flow fractionation (FFF), were made in order to quantitatively address the detection, characterization, and quantification of composite nanoparticles.

An inorganic-core/organic-shell composite particle was characterized using spICP-MS and two FFF techniques. A model Au-PS-b-PAA nanoparticle (NP) consisting of 50 nm Au NPs incorporated into polystyrene micelles was used to represent a) the formation of surface coatings on inorganic NPs and b) polymer fragments released from inorganic ENP-containing polymer nanocomposites due to material weathering or abrasion. spICP-MS analysis of the composite particle indicated that multiple Au NPs were incorporated into the PS-b-PAA micelles, consistent with transmission electron microscopy (TEM) results. The presence of multiple NPs resulted in an average incorporated Au equivalent size of 63 nm. Particle separation by asymmetric flow field-flow fractionation (AF4) provided a distribution of the composite particle hydrodynamic size (about 70-400 nm). spICP-MS analysis of the fractions provided the incorporated Au mass, which demonstrated that as the Au-PS-b-PAA NP hydrodynamic size increased, the maximum number of incorporated Au NPs (i.e., Au mass) increased. Additionally, separation by centrifugal FFF, and analysis of the fractions by spICP-MS provided the Au NP mass distributions in the Au-PS-b-PAA NPs as the mass of incorporated NPs increased.

One of the challenges to detecting ENPs in the environment is distinguishing them from NNPs. ENPs have elementally pure compositions while NNPs contain multiple elements. A bimetallic core-shell NP (Au-core/Ag-shell) was used as model for NNPs in order to develop techniques suitable for the detection of interfering NNPs. The Au-core/Ag-shell NP was first used to demonstrate how current spICP-MS capabilities (i.e., using single element analysis) in combination with AF4 can be utilized to determine the bimetallic composite nature of the Au-Ag core-shell NP. Particle number concentrations provided by spICP-MS and AF4-ICP-MS analysis showed that the particle solution contained composite NPs rather than individual Au-only and Ag-only NPs. spICP-MS was then used to observe particle dissolution in dilute acid over time to reveal the core-shell particle structure. A recent development in spICP-MS technology, dual element analysis with a quadrupole mass analyzer, offers the ability to quickly determine the composition of a particle by detecting two elements on a particle-by-particle basis within one short sample analysis time. A qualitative validation procedure with the Au-Ag NPs, Au NPs, and Ag NPs (all < 100 nm in size) showed that dual element spICP-MS detected and reported signals for the correct analytes using dwell times and settling times of 100 microseconds. A quantitative comparison between the single element mode and dual element mode average NP intensities for the Au-Ag NPs showed dual element NP intensities that converged on an average in agreement with the expected core-shell NP mass ratio. The ratio of single element NP intensity to dual element NP intensity was the same for both analytes, which may indicate an empirical factor that can be applied to convert the average NP intensity in dual element mode to a NP mass. Approximately 40% of the dual element NP peaks were false negatives for the detection of particles containing both Au and Ag. Dual element spICP-MS favors false positives for the high abundance element and false negatives for the low abundance element.
ANALYSIS OF HYDROGEN, CARBON AND OXYGEN BY GD-OES.

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Analytical glow discharges with optical (GD-OES) and mass spectrometric (GD-MS) detection are able to obtain depth resolved information about nearly all elements in solid samples, including hydrogen, carbon and oxygen, where most of the other analytical techniques fail. However, the interpretation or even quantification of the measured signals sometimes is very difficult. Effects of a physical nature (plasma processes such as the ‘Hydrogen effect’, the Doppler effect, self absorption or diffusion of hydrogen in the sample during sputtering) and a chemical nature (e.g. formation of compounds with argon or the matrix, poisoning of the sample or gettering) go along with instrumental problems (e.g. of the sensitivity and vacuum quality) and problems at calibration.

A big challenge represents the need to use calibrations samples for light elements in different matrix - especially for hydrogen and oxygen. Therefore, sintered calibration material was produced and included in fundamental investigations.

Using mixtures of TiH$_2$ and ZrH$_2$ with Cu it was found that the emission yield (EY) of H I 121.56 nm is constant up to about 0.3 m% hydrogen. At higher concentration the intensity may even reverse. It was also found that independent of the source of hydrogen - by sputtering a hydrogen containing sample or by using a gas mixture of Ar and H$_2$ – there is continuous light in the UV and VUV region emitted together with the characteristic hydrogen lines. This points to recombination and dissociation, correspondingly.

Using Al$_2$O$_3$, CuO, Cu$_2$O and MgO mixed and sintered with Cu, Al and Mg the blue line shift effect at O I 130.22 nm, first time reported by Köster 2009, was confirmed [1]. The effect is more pronounced in Mg matrix than in Al and Cu, which due to line interference leads to a matrix dependent EY. It will be shown that the effect is negligible at O I 777.19 nm and the EY is matrix independent.

For the analysis of carbon by GD-OES usually the C I 156.143 nm or C I 165.700 nm line are used. Recent investigations did show that the use of the C I 193.090 nm line must be preferred at high concentration and low sputtering rate.

DISTANCE OF FLIGHT MASS SPECTROMETRY AND NEW MODES OF TIME-OF-FLIGHT MASS SPECTROMETRY: DEVELOPING NEW ANALYTICAL CAPABILITIES THROUGH NOVEL INSTRUMENTAL APPROACHES

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Time-of-Flight Mass Spectrometry (TOFMS) is a fixture in many analytical laboratories, and it has had a particularly significant impact in the biological sciences. The reasons for this popularity are self-evident: TOFMS achieves simultaneous analysis of an unlimited m/z-range with high sensitivity (S/N) and at a very rapid spectral generation rate. Recently, we have developed several new modes of operation based on the TOFMS architecture in an effort to extend these capabilities.

The primary example is the development of a new type of mass analyzer known as the distance-of-flight mass spectrometer (DOFMS). The DOFMS concept is best explained by comparison with traditional time-of-flight mass spectrometry (TOFMS). In TOFMS, the mass-to-charge (m/z) of an ion is determined by imparting the same energy to all ions and then measuring the time required for each m/z to traverse a known distance and arrive at a single detector. In contrast, DOFMS determines the m/z of an ion by measuring the distance each ion travels over an identical time period. Simply put, ions of lower m/z traveling longer distances than ions of greater m/z, and m/z can be measured based upon ion location.

This new type of spatially-dispersive mass spectrometer therefore has new experimental requirements, such as a position-sensitive ion detector, but also provides a new set of analytical capabilities. Like TOFMS, DOFMS is architecturally simple, offers very rapid spectral generation rates, and is capable of simultaneous multielemental and isotopic analysis. Unlike TOFMS, the DOFMS is able to employ new position-sensitive solid-state array ion detectors to great advantage, providing greater detection efficiency and dynamic range than typical TOFMS. As importantly, DOFMS employs the same physical architecture as conventional TOFMS systems, and thus a combined DOFMS/TOFMS instrument can be constructed.

Through a technique we have termed ‘Zoom-TOFMS’, the DOFMS focusing strategies can also be used in a conventional TOFMS to improve mass resolution over a limited m/z-window. This approach requires no significant changes to a conventional commercial TOFMS, instead using the typical TOFMS detector for a new mode of operation. Interestingly, Zoom-TOFMS allows mass resolving power, S/N, and spectral generation rate to be increased together, something not often possible with other instrumentation. Further, as TOFMS and DOFMS share a common architecture, a single instrument is able to switch between TOFMS, Zoom-TOFMS, and DOFMS as required. The theory of operation and experimental advantages of DOFMS will be discussed, and the analytical performance of this new type of mass spectrometer coupled with atomic ionization sources will be described.
PLASMA-BASED MASS SPECTROMETRY TO CHARACTERIZE ULTRAFINE PARTICLE EXPOSURE AND REACTIVITY IN OCCUPATIONAL ENVIRONMENTS

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Exposure to airborne nanostructured particles and their associated toxicity remains in spite of numerous works this last decade an important societal concern for environmental and occupational health (diesel exhausts, ultrafine particles, fumes, manufactured nanomaterials …). Nanostructured particles can easily penetrate into the body by inhalation and reach the deepest parts of the lungs. Their small size facilitates the absorption in cells and the transfer towards the blood and lymphatic circulation to potentially reach specific organs (bone marrow, lymph nodes, spleen, heart), as well as their translocation to reach the central nervous system. The main mechanism which drives particle toxicity is their capacity to induce oxidizing stress occurring when reactive oxygen species (ROS) disrupt the balance between reaction of oxidation and antioxidizing defense. The ROS can be formed from radicals, metals of transitions or other chemical agents present on the surface of particles, or following the interaction between the particle and the cell components. There is a direct link between the surface, the capacity to generate ROS and the pro-inflammatory effects of nanostructured particles in the lung. Their higher specific surface area (SSA) compared with SSA of the same particles bigger in size makes nanostructured particles more active biologically. Besides the specific surface, other parameters of the particle can influence their toxicity, such as size, shape, chemical composition as well as reactivity and total or partial solubility of particles. In industrial hygiene, exposure to nanostructured particles is known for a long time. These particles, commonly called ultrafine particles (UFP) are secondary particles not produced deliberately. UFP are frequently associated with very exothermic processes (welding/soldering, thermal spraying, flame or laser cutting, casting) which can generate metallic ultrafine particles in very high concentrations in the air. The recent progress in particle metrology allowed a better characterization of these exposures. It is from now possible to collect these particles according to their size (from nanometers to some microns) and to determine the chemical composition as well as other parameters of interest such as speciation or bioaccessibility. A state of the art of these methods of characterization of these parameters using plasma-based spectrometry will be presented for the metallic UFP met in industrial hygiene (welding fumes, thermal spraying).
(O-9) COMPARISON OF LOAD COIL AND FLAT PLATE PLASMAS

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In the present study, robustness of load coil and flat plate plasmas were compared using SRM1633c (Coal fly ash) and SRM2782 (Industrial sludge) as test samples. Sample matrices with a high dissolved solids concentration were selected in order to find out whether the stable plasma conditions can be reached with a plasma gas flow of 8 L min\(^{-1}\) only. The low argon gas consumption enabled by the flat plate plasma technology is featured in Perkin Elmer Optima 8300. The results were compared with the Perkin Elmer Optima 4300 DV with standard load coil plasma, using plasma gas flow rate of 15 L min\(^{-1}\). A Scott-type double pass spray chamber with a cross flow nebulizer and a cyclonic spray chamber equipped with a GemCone nebulizer were tested. Sample pre-treatment methods used were microwave digestion and ultrasound-assisted digestion; the reagents used were aqua regia and hydrogen fluoride. The robustness of the plasmas was tested by measuring the Mg II 280.270 nm / Mg I 285.213 nm line intensity ratios by varying power of plasma from 1200 to 1500 W and nebulizer gas flow rates between 0.4 - 0.9 L min\(^{-1}\) at Mg concentration of 5 mg L\(^{-1}\). The most significant differences between plasmas were obtained with axial measurement resulting in Mg II / Mg I line ratios over 8 for flat plate plasma with a power of 1500 W and nebulizer gas flow rate of 0.5 - 0.7 L min\(^{-1}\) for both nebulizers.

When load coil plasma was tested, the robust plasma conditions were reached with a nebulizer gas flow of 0.4 L min\(^{-1}\) only, but with RSDs over 10 %. Thus robust plasma conditions cannot be reached for this sample matrix using load coil plasma. The emission intensities were dramatically decreased with the increasing nebulizer gas flow rate using both nebulizers.

When flat plate plasma was tested, the highest emission intensities were obtained with the nebulizer gas flow of 0.5 L min\(^{-1}\) for GemCone nebulizer, decreasing with the increasing flow rate. According to Mg II / Mg I line intensity ratios and emission intensities of ionic lines measured, the nebulizer gas flow of 0.5 and 0.6 L min\(^{-1}\) were suggested for cross flow and GemCone nebulizers, respectively. The tests showed the need of higher nebulizer gas flow for a cross flow nebulizer resulting in clearly highest emission intensities with flow rates of 0.6 and 0.7 L min\(^{-1}\).
DIAGNOSTICS AND SIMULATION OF A LOW-ARGON-FLOW ICP FOLLOWED BY OPTIMIZATION OF THE TORCH

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The LTE-based and two-temperature models are considered in view of recently obtained experimental data, which include the direct measurement of the electrical voltages and currents in the ICP load coil [1], the plasma power calculated from these values, the temperature of the torch wall, which was measured using infrared thermography, as well as rotational, excitation and electron temperatures of the plasma measured by optical methods.

The models are based on the LTE- [2] and two-temperature [3,4] models of Mostaghimi, Proulx, Boulos, Barnes \textit{et al.} and include comprehensive calculations of the wall temperature and swirl gas flows.

Both, measurements and simulations were carried out for the standard Fassel-type ICP torch, as well as for the low-argon-flow torch developed by Buscher \textit{et al.} [4-5] for a wide range of operational conditions. Satisfactory agreement with the experimentally measured electrical current in the coil, the measured plasma temperatures, and the wall temperature were found (the latter has, however, strong deviations in case of the standard torch).

The model is applied for a systematic improvement of the form of the SHIP torch to improve homogeneity of the cooling process and transport efficiency of the sample.

References


**RECENT ADVANCES IN MASS CYTOMETRY AND TISSUE IMAGING**

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**Abstract**

Mass cytometry, a technique for multidimensional analysis of single cells for multiple proteins, is based on inductively coupled plasma time-of-flight mass spectrometry. Recent developments in mass cytometry instrumentation resulted in improvements to the cell analysis rate, ease of use, sensitivity, and quality of cell-specific data. Advances in developing reagents, including cell-barcoding reagents, enable cell-staining protocols that are reliable and reproducible. Cells from up to 20 individual barcoded samples can be mixed and interrogated simultaneously by one panel of 40 or more antibodies and other metal-labeled reagents.

Through the last six years, commercially available CyTOF® systems were used for analysis of cell suspension samples. These were best-suited for detecting cells in bodily fluids, while tissue samples needed to be dissociated into single cells in order to be analyzed. We will present results of the development of a novel system based on laser ablation of tissue sections and introduction of the well-resolved single-laser-shot-generated plumes into the Helios™ CyTOF system. The method was pioneered by Bernd Bodenmiller’s and Detlef Günther’s research groups at the University of Zurich and ETH, respectively [1], which have shown simultaneous detection of 32 proteins in breast cancer tissue sections. Our new system provides enhanced performance due to the improved laser beam optical path, improved visualization optics, and improved gas dynamics of the ablation chamber. The system allows interrogation of tumor tissue sections with 1 µm spot size, at 100 Hz laser shot repetition frequency. The technique can be applied quantitatively using specially designed calibration slides.

Results of multiprotein imaging of tissue sections of human pancreatic adenocarcinoma xenografts, brain tumors, and cancer cell line samples deposited on the slides will be presented, and advances of the instrumentation discussed.

MULTI-ELEMENT BIOIMAGING OF PLANT TISSUES

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Multi-element bioimaging with Laser Ablation Inductively Plasma-Mass Spectrometry (LA-ICP-MS) is emerging as a highly versatile tool for bioimaging of plant materials, since samples ranging from <100 µm to 15 cm in diameter can be analyzed, using 1-150 µm spot sizes. Furthermore, LA-ICP-MS offers a wide range of elements to tested simultaneously and operate with a very high sensitivity, and it is more accessible than any other bioimaging technique, making it attractive for a range of purposes, including plant science. Naturally dry samples, like cereal grains, require limited sample preparation, and have therefore been frequently analyzed. The majority of plant samples, however, (leaves, stem and roots) is naturally hydrated and therefore requires special procedures for sample preparation. This talk will highlight the challenges and opportunities that are entailed by multi-element bioimaging of plant tissues with LA-ICP-MS, mainly exemplified by roots and cereal grains (representing hydrated and naturally dry samples, respectively).

Understanding the mechanisms underlying the functionality of roots is central to developing plants with root systems that are more efficient at foraging for nutrient to help drive sustainable intensification of food production and quality. In this context, bioimaging techniques can answer fundamental questions regarding ion transport that other techniques cannot. Prior to LA-ICP-MS analysis, sample dehydration must be performed without changing the tissue structure or the native composition of ions, which is a major challenge. We have developed a novel method for multi-element bioimaging of mineral nutrients and trace elements in roots of the genetic model system Arabidopsis thaliana. These improvements open up for combining targeted genetic modifications and cellular-level imaging in order to unravel the complexities of how roots selectively acquire mineral nutrient from the soil.

Cereal grains are fundamental for human health, as they constitute the major source of both macro and micro nutrients for a great number of people. Fe and Zn deficiencies, for example, affect as many as one third of the world’s population, especially the rural poor, which depend on cereal-based diets. For various reasons, rice and wheat are polished prior to consumption, which unfortunately result in net losses of >50% of these micronutrients. Therefore, biofortification strategies have emerged, aiming for Fe and Zn enrichment of the inner part of cereal grains, i.e. the endosperm. LA-ICP-MS constitute a valuable technique for validation of such biofortification efforts.

The presented methods will open a door for studying the transport and storage of most elements in plants, be it toxic elements such as arsenic (As) and cadmium (Cd), nutrients essential for plant growth, or minerals such as Fe, Zn and selenium (Se) which are essential in the humans diet. Since root analyses can now be applicable to A. thaliana, the technique opens up the use of the full range of molecular, genetic and cell biological approaches available in this system. For cereal grain analyses, LA-ICP-MS is an efficient and attractive tool for the evaluation of biofortification efforts.
LASER ABLATION - OPTICAL EMISSION SPECTROMETRY FOR ELEMENTAL BIOIMAGING

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The relevance of elemental bioimaging has increased more and more during the last years. The two-dimensional distribution of analytes in various specimen provides completely new information for biological and medical research. The samples are ablated linewise as thin cryosections by means of laser ablation and the generated aerosol is then transported into the detection system.

One of the most challenging parts of this method is the element selective detection. Typically, an ICP-MS is used for this purpose, as it is well-suited for most analytes and offers low detection limits. Most interferences can be controlled by high resolution mass analyzers or collision/reaction cell technology. However, high acquisition costs, a demanding handling and a loss of intensity in high resolution mode are major disadvantages of this approach. For this reason, elements as Ca, Na, Sr and Si are highly challenging analytes.

In this work, optical emission spectrometry is introduced as a powerful tool for elemental bioimaging. As it allow transient measurements, coupling to a laser ablation system can be realized. Particularly, alkali and alkaline earth elements show excellent detection limits and limited interferences in ICP-OES. For example, \(^{40}\)Ca is hardly detectable with a regular ICP-QMS, due to the signal of \(^{40}\)Ar and the low abundance of other calcium isotopes. The elemental distribution of Ca and Na can help to illuminate the transport mechanism of ions in plants. Further results regarding the distribution of nanoparticles can provide an improved understanding of their fate in the organism after instillation into rat lung.
IMAGING OF NANOPARTICLES IN CELLS BY LA-ICP-MS

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The interaction of nanoparticles (NPs) with cells has become a major field of interest, ranging from therapeutic applications to nanotoxicology. The cellular uptake depends on the primary characteristics of the NPs (e.g. size, shape, surface modification) and on the cells interacting with the particles. In recent years, elemental imaging of biological samples using laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) is gaining more and more importance. Latest improvements regarding spatial resolution and washout time make LA-ICP-MS particularly interesting for single cell analysis.

LA-ICP-MS was used to study the NP pathway from uptake, via intracellular processing up to cell division. Furthermore the local distribution of naturally occurring elements in cells like P was measured to indicate the cell morphology.

In our experiments, fibroblast cells and macrophages were incubated with different metal-containing NPs under varying experimental conditions. For LA analysis the cells were fixed with formaldehyde and dried. Subcellular resolution is achieved by careful optimisation of laser energy, ablation frequency and scan speed. The elemental distribution of cells incubated with NPs was determined by continuous ablation line by line.

Our results show that LA-ICP-MS is able to localise NP aggregates within cellular substructures. The studied NPs accumulate in the perinuclear region in the course of intracellular processing, e.g. multivesicular fusion and endosomal maturation, but do not enter the nucleus [1, 2]. The NP uptake depends on concentration and incubation time. Contrary to macrophages the uptake of fibroblast cells showed a strong dependence on the NP diameter indicating different NP-cell interaction [3]. Additionally, the number of NPs internalized by individual cells was determined and variations within the cell population became visible.

References
Cisplatin, carboplatin, and oxaliplatin are the most widely used class of anticancer agents used to treat many types of cancer. The mechanism of action for platinum compounds is DNA damage resulting in cell death. Initially, patients respond well to treatment but later relapse and display resistance to platinum compounds. Resistance to platinum compounds is mediated by the following mechanisms: decreased drug uptake, increased drug export, increased DNA repair, and cytosolic inactivation. This work focuses on the use of Single Cell Inductively Coupled Plasma- Mass Spectrometry (SC-ICP-MS) technique exploring the uptake mechanism for cisplatin in cells based on individual cells. Experiments were performed using the A2780 cisplatin-sensitive ovarian carcinoma cell line and the corresponding cisplatin-resistant cell line, A2780-CP70. Cells were treated with 3 µM of cisplatin and uptake was analyzed by a time course experiment 1, 2, 4, and 8 hours post-cisplatin exposure. Prior to harvesting, drug-containing media was removed and cells were rinsed three times with PBS. Cells were counted on a hemocytometer and resuspended to a final concentration of 50,000 cells/ml in cell media. Whole cell cisplatin content was measured using the Perkin Elmer NexION 350 ICP-MS. Total cellular cisplatin was measured analyzing the platinum 195 isotope using the Syngistix Application software. The Syngistix software allows the determination of platinum within each cell and creates a histogram of cisplatin uptake. The uptake of cisplatin differed between the cisplatin-sensitive A2780 cell line in comparison to the A2780-CP70 cisplatin resistant cell line. Additionally, we observed a heterogeneous distribution of cisplatin uptake in both cell lines, reflecting that drug uptake within cancer cells differs from cell to cell. Traditionally, cisplatin uptake is measured by digesting an entire cell population with nitric acid and quantitating the total platinum content by ICP-MS or other analytical technique. The downfall of this approach is it does not reflect the distribution and individual cellular variation of cisplatin uptake. Single cell analysis allows the real time uptake of cells, reflecting more of what is observed within tumor yielding the possibility to determine resistance status. Additionally, SC-ICP-MS allows for the development of experimental models to determine drug delivery and efficacy translating to a better response in the clinic.
QUANTITATIVE BIOIMAGING BY MEANS OF LA-ICP-MS REVEALS SILVER MIGRATION FROM SILVER-COATED ENDOPROSTHESSES INTO THE PATIENT’S BRAIN

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Osseous defects, which result from the removal of a tumor or metastasis, are reconstructed with mega-endoprostheses. Especially in case of oncological patients, post-operative infection rates of up to 35% occur, often followed by revision surgery or, in the worst case, by amputation of affected extremities. Alternatively, silver is used as implant coating due to its antimicrobial properties to prevent bacterial infection. However, besides killing germs, silver also affects human cells when exceeding a cytotoxic concentration level. Since information about the fate and a possible accumulation of the released silver in the organism is limited, our present work focusses on laser ablation-based elemental bioimaging of brain as possible target organ of patients with silver-coated endoprostheses.

In detail, thin brain sections of a patient with three silver-coated endoprostheses (total Ag introduction of 2.9 g) are investigated. Quantification of silver in five brain areas, including cerebellum, medulla oblongata and basal ganglia, is performed using matrix-matched standards, revealing a hundredfold increased silver concentration compared to native brain tissue of patients of the same age and sex. Additionally, high resolution LA-ICP-MS with a spot size of 4 µm is performed to allocate the silver deposits to relevant cell types. Therefore, an immunohistochemical stain with a gold-labelled secondary antibody is implemented. Overlays of the elemental distribution images of both $^{107}$Ag and $^{197}$Au enable the evaluation of the correlation of silver signals to marked cell type. Besides an accumulation in endothelial cells of blood vessels, silver can be co-localized to neurons and glial cells, demonstrating its ability to cross the blood-brain-barrier. Thus, elemental bioimaging by means of LA-ICP-MS empowers to obtain an improved understanding of transport, distribution, and deposition of silver in biological tissues.
QUANTITATIVE BIOIMAGING OF PLATINUM VIA ON-LINE ISOTOPIC DILUTION-LA-ICP-MS

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Platinum-based anti-cancer drugs play an important role in modern chemotherapy. Cisplatin [cis-diamminedichloroplatinum(II)] is the most commonly applied compound due to its unmatched curing chances for certain types of cancer like testicular, ovarian, bladder and lung cancer. The visualization of elemental distributions in biological tissues may provide valuable information about biological and medical correlations, thus the need for spatially resolved and quantitative information is increasing. Often, trace concentrations are encountered and therefore, sensitive and reliable quantification methods (LA-ICP-MS) are required. The most common quantification strategy is external calibration, which includes matrix matching of the standards. However, the preparation of matrix-matched standards is laborious and sometimes homogeneous distribution of the spiked element and similar behaviour of the standard tissue and the specimen is difficult to achieve. An alternative powerful quantification approach is isotopic dilution analysis (IDA). IDA is an absolute quantification method, which is generally traceable to SI units and less affected by drift effects and statistical errors compared to external calibration. Generally, the analyte and the corresponding isotopically enriched spike can be mixed off-line or on-line, where the achievement of the isotopic equilibrium is essential. Within this work, the combination of on-line IDA and bioimaging based on LA-ICP-MS is presented. Therefore, the method was applied to rat kidney samples from Cisplatin perfusion experiments. The results show a high accordance with the conventional external calibration approach and demonstrate the applicability of the developed on-line IDA method for elemental bioimaging.
(O-18)

MEASUREMENTS OF ISOTOPE ABUNDANCES IN LASER PLASMAS

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Laser ablation is an excellent direct solid sampling technology for rapidly measuring the elemental and isotope content of any material. The laser pulse duration and energy drives the ablation process, which converts a portion of the sample into a luminous optical plasmas that condenses to a fine aerosol. Chemical analysis is instantaneous by measuring the emission spectra in the optical plasma, or by transporting the aerosol to a secondary source for excitation and analysis. The physics of the laser matter interaction influences chemical analysis capabilities, including range of elements detected, sensitivity of converting laser photons to ablated mass, and accuracy and precision of analysis. Traditionally, laser ablation sampling has been coupled with ICP-MS (Inductively Coupled Plasma Mass Spectrometry) for sensitive isotopic analysis. Alternatively, optical emission from the luminous plasma initiated by the ablation process at the sample surface can be monitored and provide elemental analysis and in some cases isotopic analysis. This well-known optical approach is called LIBS (Laser-Induced Breakdown Spectroscopy).

LIBS is ideal for analyzing light elements, for example Li, Be, S, C, O, N, H and the halogens; elements that are difficult or impossible with the ICP-MS. In addition LIBS is good for major concentrations which again are problematic in the ICP-MS, expanding the dynamic range of analysis. These two detection modalities complement each other nicely. To go one step further is to measure the isotopes of these light elements and halogens. However, splitting in atomic and ionic spectra from isotopes in atmospheric pressure laser plasmas is generally small and poorly resolved.

Our new technology LAMIS (Laser Ablation Molecular Isotopic Spectroscopy) measures isotopes in laser plasmas at atmospheric pressure from molecular emission band spectra in addition to measuring atomic and ionic line spectra. The time-resolved dynamics of the plasma allow both line and band spectra from every laser pulse. Molecular isotopic shifts are orders of magnitude greater than atomic and ionic transitions. By measuring molecular emission spectra as the plasma cools, isotopic spectral splitting is enhanced up to several orders of magnitude. We developed LAMIS to date by measuring B, C, H, D, Sr and other isotopes. We demonstrated low percent levels for sensitivity and have experimental plans to meet ppm levels. For some isotopes, we have achieved < 0.1% precision. The talk will describe the isotope work that has been reported in laser plasmas at atmospheric pressure and show how LAMIS expands the capabilities of laser ablation for measuring every element/isotope on the periodic chart for each laser pulse.

References:
(O-19)
VARIABILITY IN TRACE ELEMENT ISOTOPE COMPOSITION IN ENVIRONMENTAL MATRICES

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High precision isotope ratio measurements by ICP-SFMS and MC-ICP-MS found increasing applications in environment studies allowing tracing sources and processes using stable and radiogenic isotope signatures. The usefulness of such approach can be limited by analytical challenges in obtaining reliable isotope information (especially for elements present at trace levels relevant for uncontaminated environmental matrices or in situations where available sample size is limited) and by natural variability of isotope compositions. The later was assessed for several isotope systems (Pb, Sr, Os, B, Cr, Fe, Cu, Zn, Ag, Cd, and Tl) and in various environmental samples (soil profiles, lisimetric water, leaves, needles, mushroom, lichen, mosses) from Northern Sweden.
DETERMINATION OF LONG-LIVED RADIONUCLIDES AT SUB NG.KG-1 LEVELS IN PRESENCE OF HIGH AMOUNT OF ISOBARIC INTERFERENCES BY ICP-QQQ-MS

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Long-lived, high abundance radionuclides are of increasing environmental interest with regards to decommissioning of nuclear sites, and longer term nuclear waste storage and disposal. Traditionally, alpha and beta counting techniques are used for the measurement of these radionuclides but these techniques are not feasible to detect the low levels present in environmental samples. The long half-life, low specific activity and presence of other radionuclides makes them very difficult to measure with radiometric methods even at higher levels in nuclear waste materials. However, inductively-coupled plasma mass spectrometry (ICP-MS) has been shown to be an attractive tool for long-lived nuclides quantification1, 2 but the presences of isobaric interferences have prevented the application of this technique for their accurate quantification. Zirconium-93 is a pure beta-emitting radionuclide that can be determined by liquid scintillation counting, which decays to metastable 93Nb (half-life 16.1 years) to give stable 93Nb. The long half-life of 93Zr (1.64 x 10^6 years) will contribute significantly to the activity of the long-term nuclear waste repository, and in 1,000 years, 93Zr will be the second largest contributor to fission product activity3. ICP-MS can be used to accurately determine the 93Zr content in a range of matrices but the inevitable presence of 93Nb affect its accurate determination. In this work, we suggest a novel approach for method development to achieve accurate determination of 93Zr in the presence of isobaric interferences, using stable isotopes and triple-quadrupole ICP-MS capabilities. The approach relies on the potential of reaction cell chemistry for isobaric interferences removal by using variety of cell gases. The developed method offers ultimate selectivity (up to 5 orders of magnitude alleviation of the isobaric 93Nb and 93Mo) providing unbiased results and unmatched low limits of quantification for 93Zr (1.3×10^5 Bq/g or 0.14 pg/g) in environmental samples, opening new horizons to the radionuclide industry and environmental control agencies for monitoring, legislation and further efficient radionuclides environmental control. Scoping studies for the evaluation of the metrological characteristics of the newly developed method have already been performed, including initial experiments for the determination of the full measurement uncertainty. Promising results have been obtained, suggesting the methods can be further used for standardisation of 93Zr solutions, along with the existing radiometric methods. The measurements with ICP-QQQ-MS can also contribute to more accurate half-life determinations to benefit the radionuclide industry.

2 Amr, M.A. The collision/reaction cell and its application in inductively coupled plasma mass spectrometry for the determination of radioisotopes: A literature review. Advances in Applied Science Research 3 (4) (2012), 2179-2191
ISOTOPIC FRACTIONATION OF NATURAL URANIUM IN A HUMAN NEURONAL CELL MODEL: A PROOF OF CONCEPT

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The study of isotopic fractionations of metals has recently been identified as a possible new biomarker for the investigation of metabolic processes, and as a very promising tool for the early diagnosis of diseases such as cancer or neurodegenerative disorders. In vitro isotopic fractionation studies in cell cultures display an outstanding potential to help in understanding biological processes governing isotopic fractionation, and eventually to help identify cellular pathways for metals. However, the use of cellular models for these types of investigations has been under-exploited so far.

With the aim of investigating uranium cellular pathways and potential toxicity mechanisms during chronic exposure to non-toxic amounts, human cells differentiated into neurons were exposed to non-toxic natural uranium concentrations for seven days. The isotopic fractionation of uranium during its uptake by cells was studied. Given the small size of these samples, the low amounts of U incorporated by cells, and the complexity of the matrices, efforts were initially focused on the development and validation of a fit-for-purpose analytical procedure for U isotope ratio measurements. A highly efficient micro-flow sample introduction system working at 10 μL/min was used, allowing the determination of δ238 values for the ratio \( n(^{238}U)/n(^{235}U) \), with an uncertainty of ~0.1‰ for uranium amounts as little as 20 ng. An isotopic fractionation was observed for intracellular uranium, the lightest isotope \( ^{235}U \) being preferentially incorporated by cells. These results seem compatible with processes of uranium uptake such as passive diffusion according to a mass dependent fractionation, or with the incorporation via a transport protein and a mass-independent nuclear volume effect. The homeostatic modulation of endogenous elements (P, Mg, Ca, Fe, Cu, Zn, Mn, Rb) induced by uranium accumulation will be discussed as well, since it can provide clues for the identification of uranium incorporation routes and potential toxicity mechanisms.

Results of this work demonstrate the great potential of in vitro studies in cellular models as a tool to help identify cellular pathways of metals and for the understanding of processes leading to isotopic fractionations in living organisms. This information would be of prime importance in the framework of toxicological studies, as well as for the interpretation of clinical results in case of contamination or diseases.
DETERMINATION OF IRON, NICKEL AND VANADIUM IN PETROLEUM HEAVY OIL AND RESIDUE SAMPLES BY MP-AES

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Atmospheric and vacuum residue petroleum samples are very difficult samples to characterize. Traditionally, density and viscosity measurements are the dominant, however, the availability of knowing hydrogen, nitrogen, carbon and metals (besides sulfur) are becoming critical in the refining business.¹

In the past, metal analysis has been done by acid digestion followed by spectroscopy techniques such as Flame Atomic Absorption (FAA) or by plasma techniques such as Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) and Inductively Coupled Plasma Mass Spectrometry (ICP-MS). In this study we present MP-AES as a useful tool for determining Fe, Ni, and V in these heavy residue samples. Samples evaluated covered APIs from 7–38° among others properties. Determination of the metals in heavy oil and residues was evaluated comparing traditional detection techniques such as ICP-AES with MP-AES. Two sample preparation methods, wet acid digestion and direct dilution in an organic solvent were also compared.

Ten residue samples were selected according to API as well as elemental composition; API values spanning from -2° up to 18°, elemental composition as follows: S (1.0 at. % up to 5.5 wt. %) and N (200 ppm up to 14000 ppm). An Agilent 4200 MP-AES (Agilent Technologies, Santa Clara, CA, USA), with an Agilent 4107 nitrogen generator (Agilent) was used for the direct injection of o-xylene.

Excellent agreement among all elements can be obtained using both plasma gas sources. Nitrogen sustained gas for plasma can be effectively used to analyze residue samples by direct dilution method.

![Figure 1](image)

From Figure 1, it can be seen that nickel, vanadium, and iron displayed R² values above 0.95 which is very well accepted for analysis of hydrocarbon samples. Direct dilution in combination with nitrogen sustained plasma (MP-AES), provides a real safe and economical alternative to the metal analysis on residue samples.

References

RARE EARTH METALS IN BIOMASS FLY ASH – ANALYSIS USING ICP-OES

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Rare earth metals (REMs) have recently been recognized as critical materials for the European Union. With no production in the EU currently, there is a need to look further for these important metals. Simultaneously, fly ash is being produced annually in huge amounts, the disposal of which can be difficult and costly. If the concentrations of REMs in fly ash are high enough, recovering them could prove to be a cost-effective way of both disposing the fly ash and acquiring these critical materials.

The question then becomes: how to determine low ppm levels of REMs in a fly ash matrix containing several mass percentages of aluminum, iron, calcium and silicon? One of the most crucial challenges faced in determination of REMs using ICP-OES can be severe spectral interferences, caused by matrix elements and the more abundant REMs. The presence of these interferences was studied, and it was found that iron and calcium were the most common causes of interferences. High resolution and spectral profiling available in PerkinElmer Optima 8300 were used to identify overlaps in the spectra (figure 1). Using high resolution, at least one interference-free wavelength was found for each element, enabling accurate results. Optimum sample introduction and plasma conditions were also studied to reach robust plasma and highest intensities: instrumental detection limits were in the range of 0.05-1.6 μg l⁻¹. ICP-OES hence proves its effectiveness in REM analysis from a difficult sample matrix. Most REMs were enriched in Finnish biomass fly ash samples compared to crust, with the highest total concentration of 560 mg kg⁻¹. Hence, the current work introduces biomass fly ash as a feasible source for REMs, and gives rise to studying concentrations of REMs further from a wider range of fly ash samples.

Figure 1. Spectra of a fly ash sample measured using normal resolution (green) and high resolution with spectral profiling (pink). The interference of cerium on a neodymium peak is visible only using high resolution.
TRANSPORT OF TOXIC METALS TO NORWAY 1976 – 2016

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Inspired by results from “acid rain” research in southern Norway and work by Swedish colleagues using mosses for monitoring atmospheric metal deposition, we started 1976 research on trans-boundary atmospheric supply of metals to Norway. A network of 470 sites all over Norway were selected for moss sampling, and since 1985 this has been a regular activity of the national monitoring program for air pollutants. This activity has been supported by studies of metals in aerosols and precipitation samples, and calibration exercises have been carried out to convert metal concentrations in moss to bulk deposition rates of the same elements. The time axis of metal deposition is extended to past centuries by analysis of peat cores from ombrotrophic bogs.

Initially much higher atmospheric deposition rates of several elements, such as Pb, Zn, Cd, As, and Sb, were observed in the southernmost parts than in other parts of the country, due to long-range atmospheric transport of polluted air from areas elsewhere in Europe. These deposition rates have decreased substantially with time all over the country. As an example the present Pb deposition in the far south is only around 5% of the level observed 40 years before, and similar reductions have been observed for other volatile metals. In the far northeast the opposite trend is observed in the trans-boundary supply of Ni and Cu from Russian metal smelters, where a substantial increase has occurred relative to the levels 40 years ago.

Collaboration with scientists from The Norwegian Institute for Air Research over the entire time period is gratefully acknowledged.
(O-25)
QUANTITATIVE DETERMINATION OF MAJOR- MINOR- AND TRACE ELEMENTS IN BEER USING ICP-OES SPECTROMETRY

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Beer is the most popular alcoholic beverage in Europe. In Germany, beer enjoys a particularly high status due to the German Beer Purity Law of 1516 (the “Reinheitsgebot”), which uniquely defines the ingredients of beer to be hop, malt, yeast and water. This makes the German Beer Purity Law the oldest food law in the world, which is still valid today and makes beer, in addition to drinking water, one of the most researched food products with the highest standards regarding quality, freshness, appearance and flavor. Statistically, per capita beer consumption in European countries was 68 liters in 2013. The highest per capita beer consumption was determined for the Czech Republic (144 L), followed by Germany (107 L) and Austria (106 L). For these levels of consumption, the question arises: just how healthy is beer and what does beer contain?

In accordance with the German Beer Purity Law of 1516, German beer contains the ingredients hops, malt, yeast and water, as well as all major B vitamins. Vitamin B2 and B6, which are important for metabolism, are particularly abundant in beer. In addition, bitter substances and essential oils are indisputably effective against loss of appetite, gastric disorders and states of anxiety. Japanese scientists have found active ingredients in 24 types of beer that inhibit so-called heterocyclic amines and thus the onset of cancer. This has been confirmed by German scientists, who see the same tumor-preventing effects of the polyphenolic flavonoids as are claimed for red wine. Last but not least, beer is diuretic, and its phenolic ingredients support the cardiovascular system. In addition, beer contains minerals and trace elements (e.g. Ca, Na, Mg and Zn) that are important for human nutrition. However, undesirable substances such as heavy metals (for instance Cd, Pb, Hg and As) are also found.

For simultaneous multi-element analysis, an atomic emission spectrometer with an inductively-coupled plasma like the ICPE-9820 with vertical minitorch position and ‘dual view’ (axial and radial) plasma observation has been used. This method enables the analysis of samples with low concentrations of just a few µg/L (axial) such as Pb, Cd, Mn, Hg, As and Sb and high concentrations of approximately 0.5 mg/L up to 150 mg/L (radial) such as Na, K, Ca and Mg within a single analysis sequence. Implementation of this method is carried out in accordance with DIN EN ISO 11885 (2009). The beer samples were degassed for 5 minutes in an ultrasonic water bath and subsequently diluted with water to 1:1 and 1:5 respectively. Calibration was performed against aqueous standard solutions. Analytical results for a variety of beers are presented.
(O-26)

SEQUENTIAL EXTRACTION OF POTENTIALLY TOXIC ELEMENTS IN SEDIMENTS AND SPECIATION OF ARSENIC IN SEDIMENTS AND WATER

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The levels of potentially toxic elements and their species are important considerations in the safety and quality of drinking water. Supply of potable water is challenging, particularly in the rural areas of South Africa where poverty levels are high. Inadequate supplies of potable water in these areas often results in poor households having no option but to use water from unsafe sources. The aim of this study was to assess the levels of potentially toxic elements and their species in water and sediment samples collected from Mokolo and Blood Rivers in Limpopo province, South Africa. Sediment and water samples were collected from these rivers. The microwave assisted sequential extraction procedure was evaluated to obtain extraction efficiencies similar to the European Standard, Measurements and Testing (SM & T) program procedure, in less time, while using smaller volume of reagents. The method was validated using BCR 701 certified reference material for sediments. The levels of V, Mn, Pb, Cd, Ni, Cu, Fe, Zn, Cr and As in sediment and water samples were quantified using inductively coupled plasma-mass spectrometry (ICP-MS). In particular, chemical speciation of As in water and sediment samples was conducted using high performance liquid chromatography (HPLC) coupled to ICP-MS. This study will assist the Department of Water Affairs and Department of Health to evaluate the potential health risks that rise from the consumption of contaminated water.
STATE OF THE ART AND PERSPECTIVES OF PLASMA-BASED MASS SPECTROMETRY TECHNIQUES FOR DIRECT SOLID ANALYSIS AT THE MICRO AND NANOSCALE

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The combination of a plasma-based ionization source allowing highly spatial resolved (i.e. lateral and/or in-depth) direct solid analysis with mass spectrometry (MS) results in powerful analytical tools offering elemental, isotopic and molecular information for the characterization at micro and nanoscale levels. Through representative examples of applications carried out in our laboratory, in this presentation it will be reviewed advances in two powerful direct solid analysis plasma-based MS tools that can be considered as complementary in terms of spatial resolution achievable: glow discharge (GD) - mass spectrometry (MS) for thin film analysis and laser ablation (LA) coupled to ICP-MS for imaging purposes.

The coupling of radiofrequency pulsed glow discharges (RF-PGDs) to time of flight mass spectrometry (TOFMS) provides an amazing amount of data because the TOFMS time-gated detection permits to collect complete mass spectra produced by different ionization mechanisms within each single GD pulse. This emerging technique makes possible to obtain multielemental depth-profiles with high depth resolution, isotope ratio measurements, as well as the simultaneous production of elemental, structural and molecular information. Considering the promising application field of RF-PGD-TOFMS, further research is still welcomed to achieve the best performance of this direct solid analysis tool. In this context, the addition of helium and oxygen to the argon discharge gas will be evaluated in this presentation. The analytical potential of RF-PGD-TOFMS will be shown through an overview of recent applications.

The quantitative analysis and distribution of essential, toxic, and therapeutic metals, as well as metalloids and nonmetals in biological tissues is a key task in life sciences today. In this vein, LA-ICP-MS has been established as a promising powerful and sensitive multielement technique for the quantitative determination of trace elements in biological materials. In this presentation, capabilities of LA-ICP-MS for elemental bioimaging of very different types of tissues (e.g. rice, green leaves and human tissues) will be shown. Through these representative examples it will be discussed the problems related with the reliable quantitative analysis of heterogeneous samples and strategies developed in our laboratory for such purpose will be presented.
ANALYSIS OF HARD-TO-DIGEST CERAMICS BY USING LASER ABLATION IN LIQUID.

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Laser Ablation in liquid (LAL) sampling was used to generate micro- to nanosized particles from sintered SiC and single-crystal SiC. LAL sampling has two advantages: it generates particles smaller than several micrometers, and it transforms hard-to-digest crystals to soluble spherical particles. This allows LAL-sampled micro- to nanoparticles to easily decompose by acid digestion owing to the increase in surface area resulting from transformation of the particles by the melting–congelation process that occurred during laser ablation of the sample surface. After acid digestion, the LAL-sampled particles were analyzed by inductively coupled plasma mass spectrometry (ICPMS). Because solution calibration can be used with LAL-ICPMS, this technique provides more-accurate data than direct solid analysis by means of laser ablation–ICPMS, secondary ion mass spectrometry, or glow discharge mass spectrometry. Here, we demonstrated for the first time that LAL sampling can be used to determine trace elements in hard-to-digest samples (sintered SiC and single-crystal SiC). Results obtained by LA-ICPMS and LAL-ICPMS were compared in terms of accuracy and limit of quantification. In the sintered SiC Ti, Cr, Fe, Co, Ni, Sr, and W impurities were found to be homogeneously distributed, whereas the surface layer was contaminated by Al, Mn, Cu, Y, and Zr. In the single-crystal SiC Al, Ti, Cr, Mn, Fe, Ni, Cu, and Zr impurities were detected. The limits of quantification of LAL-ICPMS were 0.1–1.0 µg g⁻¹ for Al, Ti, Cr, Mn, Fe, Co, Ni, Cu, Sr, Y, Zr, and W.
ICP-TOFMS FOR COMPREHENSIVE ELEMENTAL ANALYSIS OF DISCRETE SAMPLES: APPLICATIONS IN LASER ABLATION AND SINGLE-NANOPARTICLE ANALYSIS

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ICPMS instruments have gained widespread use for elemental analysis because they provide—among other characteristics—low detection limits, broad dynamic range, and multi-isotope analysis. However, for a range of emerging applications, such as single-nanoparticle detection or high-resolution elemental imaging, the analysis speed of conventional ICPMS systems, which analyze ions of different mass-to-charge values sequentially, is limiting. For example, in single-nanoparticle ICPMS (spICPMS), the transient signal of a single NP is about 300 µs. Over this short duration, conventional quadrupole- or single-collector sector-field-ICPMS instruments can only provide abundance information for one (or at most two) isotopes. However, the monitoring of only one isotope per NP is disadvantageous because the detection of “fingerprint” elements or isotopes in NPs is one of the most promising ways to differentiate between naturally occurring and human-made particles, and thus is of great importance to the study of NPs in the environment. To overcome the limitations of sequential ICPMS instruments for transient analysis, a clear solution is to employ simultaneous full-spectrum mass analyzers such as time-of-flight mass spectrometers (TOFMS) or Mattauch-Herzog mass spectrographs (MHMS). Here, we support the argument for simultaneous mass analysis in ICPMS with recent results from a newly developed ICP-TOFMS instrument.

Specifically, we will describe how high-speed simultaneous multi-elemental detection with ICP-TOFMS has been employed for the detection of engineered CeO₂ NPs in a background of Ce-containing natural nanomaterials. Moreover, through a combination of standard pneumatic-nebulization sample-introduction with discrete-droplet introduction of standard solutions, we demonstrate a method for online size calibration and instrument-drift correction for the analysis of multi-element mixtures of NPs. Finally, we will report recent progress in the combination of low-dispersion laser ablation (LA) to ICP-TOFMS. With this setup, we leverage the high-speed (up to 1000 spectra/sec) and full-element detection capabilities of ICP-TOFMS in order to generate shot-resolved, high-resolution LA-ICPMS elemental images at image acquisition rates of 20 pixels/sec. The figures of merit and application of this elemental imaging approach will be discussed.
(O-30)
A TIME RESOLVED STUDY OF THIN COATED AND MULTI-LAYERED COMPOSITE MATERIALS USING THE ASTRUM GDMS

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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 bulk materials. By means of a flat geometry GD source configuration, it is also a proven technique for high sensitivity depth dependent distribution analysis of trace elements in coatings and other multi-layered advanced materials. The Astrum, the latest generation in HR-dc-GDMS technology from Nu Instruments, is a perfect candidate for such time resolved analysis. Due to the low-flow GD source of the instrument, the Astrum at normal analytical conditions (1kV,2mA) has a sputter rate which can range from 1 µm / 2 minutes for copper to 1 µm / 6 minutes for ceramics. If operational conditions are lowered, the sputter rate can be slowed by a least 5x. This low sputter rate translates into ample time to acquire meaningful and reproducible data for thin and even ultrathin films, layers and coatings. The low sputter rate also allows for time resolved analysis of coated powders as well as bulk composite materials, while obtaining information about the contaminants in each matrix layer. The Astrum offers some significant advantages over other instrumentation frequently used for depth profile work. For example, as the Astrum does not rely on matrix matched standards, the time needed and possible errors generated for this matrix dependent step are eliminated. Secondly, as the RSF values are matrix independent, the same standard relative sensitivity factor (StdRSF) data set can be used throughout each time dependent study to achieve semi-quantitative results directly. Thirdly, as the Astrum offers both low power and cryo-cooling, temperature sensitive materials can be readily analysed and interferences from high gas backgrounds are significantly reduced. Lastly, as the Astrum is capable of analysing all types of materials, composite samples containing layers of both conducting and non-conducting material can be evaluated. The study presented here takes a closer look at the depth profiling capabilities of the Astrum by evaluating the thin coating of a powder sample and the layers of a computer hard disk platter. For both materials, the matrix for each layer along with its trace contaminants are evaluated and discussed. The matrix independent RSFs, the low sputter rate and the cryo-cooling of the source are among some of the many features which serve to make the Astrum a powerful and versatile technique in the evaluation of the layers in conductive, semi-conductive and non-conductive composition materials.
(O-31)
SPECIATION ANALYSIS WITH ICP-MS DETECTION IN DRUG DEVELOPMENT

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ICP-MS is an attractive alternative to conventional detection methods like UV-spectrophotometry and electrospray mass spectrometry used in drug development. Besides the high selectivity and sensitivity, the advantage of ICP-MS detection is the linearity, which often covers several orders of magnitude and the – in principle – species independent sensitivity of the technique. However, the use of ICP-MS detection is limited to drugs containing either a metal or a hetero-element like P, As, Sb, S, Se, Cl, Br, or I.

In drug development studies, efficient separation methods are needed to separate the active pharmaceutical ingredient (API) from excipients in drug stability studies or from the complicated biological matrix like body fluids and tissue extracts when pharmacokinetic studies are performed. The high selectivity of ICP-MS detection offers the possibility of detecting very low concentrations of the API in presence of complicated matrices and the species independent sensitivity allows for detection and quantification of unidentified degradation products or metabolites in the early drug development phase.

Examples of analysis of drugs by liquid chromatography (LC) and capillary electrophoresis (CE) coupled to ICP-MS detection will be given with focus on the advantages and limitations of using ICP-MS as detection method.

Characterization of liposome formulations of platinum-based drugs by CE-ICP-MS by simultaneous measurement of Pt and P in stability studies and the release of API from the liposomes in plasma will be described.

Analysis of a pharmaceutical peptide model compound by LC-ICP-MS by detection of selenium after Se labeling and attempts to quantify peptides by detection of sulfur in plasma samples are other examples.
ICP MS-BASED TECHNIQUES TO TRACK FOR URANIUM MOLECULAR TARGETS IN REPRODUCTIVE ORGAN OF ZEBRAFISH

Sandra Mounicou

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Uranium (U), naturally occurring or anthropically released in the environment, is a key element to study because of its chemical and radiological toxicity leading to deleterious biological effects. Particularly, chronic exposure of zebrafish (Danio rerio) to low doses of uranium led to high level of U in gonad and a decrease of the spawn frequency of female. One hypothesis would be the interaction of uranium with vitellogenin (the dominant protein of oocyte and essential for the embryo development) impacting thus functionality of this protein. Therefore to improve the understanding of uranium toxicity mechanisms and metabolism, the verification of this hypothesis and the identification of others uranium molecular targets (notably proteins), is a key step.

UO$_2^{2+}$ mainly found in aerobic media, is able to link oxygen and nitrogen atoms of biomolecules to form non covalent complexes. Thus identifying uranium-protein complexes, remains challenging because of the electrostatic nature of the binding and their low abundance in a complex biological environment. These criteria imply the use of powerful analytical methodologies gathering a highly resolving separation by liquid chromatography and/or electrophoresis with a sensitive detection by elemental (ICP MS) and in parallel molecular (electrospray MS/MS) mass spectrometry.

This communication presents analytical methodologies developed for the investigation of uranium-protein complexes in gonad cytosol of waterborne uranium exposed female zebrafish. The potential of non denaturating separation mechanisms in liquid phase (Size Exclusion Chromatography (SEC) and off gel Isoelectric Focusing (IEF)) is explored. The probing of uranium-proteins entities as well as the presence of others endogenous elements (P, Fe, Cu, Zn) is directly achieved by ICP MS in liquid phase. The SEC-ICP-SFMS study demonstrates the binding of uranium with 4 different molecular weight fractions with a majority (c.a 90%) of uranium bound to biomolecule(s) in the 40 kDa range co-eluting with phosphorus and iron. The separation of uranium-protein complexes according to their isoelectric point (IEF) shows the focusing of uranium mainly in basic fractions (pH 7-8). According to these data, one of these proteins could be a fragment of vitellogenin; the formal identification of these targets will be carried out using proteolytic digestion protocol in conjunction with Reversed Phase Liquid Chromatography coupled to molecular Mass Spectrometry (µRPC-ESI MS/MS).

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SPECIATION ANALYSIS AND ELEMENT-BIOIMAGING: FROM GENETIC DISEASES TO NEW IMPLANTS

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The analysis of metals and metalloids in their different oxidation states and binding forms that means their chemical species is of paramount importance, since the properties and characteristics of the metal compounds do not primarily depend on the metal content but on the individual characteristics of the species. Thus, the acute toxicity of arsenobetaine, the main arsenic species found in fish is of the order of table salt while arsenate at the same concentration is highly toxic. Therefore analytical strategies and methods for measuring the concentration of metal species in complex samples such as biological and environmental materials are required. Such strategies and methods shall be discussed in this presentation using two examples, the investigation of metal release from implants and the copper accumulation in patients suffering from the Wilson’s disease.

Wilson’s disease is an autosomal recessive genetic disorder in which copper accumulates in tissues. One of the possibilities for diagnosis is the determination of the copper concentration in liver biopsy samples from the patient. For this purpose, methods for mapping the copper concentration in thin tissue sections were developed. Laser ablation coupled with inductively coupled plasma-mass spectrometry (LA-ICP-MS) was used for imaging with a spatial resolution of a few µm.

The second part of the presentation will discuss the release of metal species from implants and their distribution in the organism: Metal implants may release ionic species of their main components or coatings under mechanic stress which are able to form adducts with proteins and other biomolecules. Such species can be identified by using a combination of high-performance liquid chromatography (HPLC) with electrospray mass spectrometry (ESI-MS) and can be quantified by using the complementary ICP-MS.
SPECIATION OF ORGANOPHOSPHATES AND FLUOROPHOSPHATES IN LITHIUM ION BATTERY ELECTROLYTES

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Lithium-ion batteries (LIBs) are the fundament of modern consumer electronics and portable devices. Furthermore, these energy storage systems are the most promising technology for electric and hybrid electric vehicles. Unfortunately, the lifetime of LIBs is limited due to electrochemical, thermal and calendric aging. Currently applied electrolytes for LIBs usually consist of LiPF$_6$ as conducting salt dissolved in a mixture of different linear and cyclic organic carbonates, e.g. ethyl methyl carbonate (EMC) and ethylene carbonate (EC). Despite the chemical and thermal instability in organic carbonates towards the P-F bond, LiPF$_6$ is the most commercial applied conducting salt today. However, due to its instabilities, hexafluorophosphate undergoes the decomposition to the highly reactive PF$_5$. As a consequence, numerous decomposition products are formed and are an ongoing subject of investigations with different methods. The variety of decomposition products ranges from HF, inorganic and organic phosphates (OPs), dicarboxylates, diols and alkyl fluorides.

Since several of the identified decomposition products contain a P-F bond in their structure, there is a structural resemblance to certain nerve agents. The question about the potential toxicity of the used LIB electrolytes and their decomposition products is important as the improvement of the cycle life. The toxicity of Organophosphates is because of their reaction with the enzyme acetylcholinesterase (AChE). AChE hydrolyses acetylcholine, a neurotransmitter regarding muscles and organs. During the reaction, a covalent bond between the organophosphate and the AChE is formed and subsequently stopping the enzyme from functioning. Therefore, investigation of LIB electrolyte decomposition products in terms of qualitative and quantitative information is not only important for the electrochemical life time, but as well for potentially toxicological aspects. So far, only few information about the toxicity and even fewer about quantitative data are available in literature.

In this work, we present our approach to obtain structural and quantitative information about the decomposition products, especially the potential toxic compounds, by combination of inductively coupled plasma mass spectrometry with different chromatographic methods like ion chromatography or gas chromatography.
A NOVEL APPROACH FOR SPECIATION OF CR$^{3+}$ AND CR$^{6+}$ IN TOTAL PARTICULATE MATTER FROM MAINSTREAM TOBACCO SMOKE UTILIZING ETV-ICP-MS.

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Cigarette smoke is composed of approximately 6000 constituents in the gas and particulate phases and its composition is complex and rich in organics. Trace metals comprise a very low percentage of mainstream smoke and chromium an even lower fraction of trace metals present. Nevertheless, chromium has been reported to accumulate in the lungs of smokers with a biological half-life of several years.

We have previously reported total chromium concentrations in Total Particulate Matter (TPM) from mainstream smoke as below the method LODs (< 1 ng/cig) in two smoking regimens. A novel approach to speciation of Cr$^{3+}$ and Cr$^{6+}$ in TPM at extremely low levels is presented here. Speciation of Cr$^{3+}$ and Cr$^{6+}$ and quantitation of total chromium in TPM at these low concentrations must incorporate highly sensitive hyphenated instrumentation. Because of the necessity for a highly sensitive analytical technique and the removal of high levels of on-mass polyatomic interferences, mainly argon carbide (40Ar12C$^+$) and argon oxide (36Ar16O$^+$) amongst other interferences that form in the plasma, Electrothermal Vaporization-Inductively Coupled Plasma-Mass Spectrometry (ETV-QQQ-ICP-MS) was chosen. This technique utilizes CCl$_4$ saturated argon as a reactive gas in ETV for greater sensitivity and NH$_3$ as the reactive cell gas in QQQ-ICP-MS to ‘mass shift’ analyte ions away from their polyatomic interferences by forming the diammine complex ions. Species specific double spike isotope dilution was investigated for quantification of Cr$^{3+}$ and Cr$^{6+}$ because of the transformations that occur prior to or during analysis. Single spike isotope dilution was utilized for quantitation of total chromium in TPM.

Sample cigarettes were conditioned according to ISO 3402 prior to smoking, then smoked according to Health Canada Intense smoking parameters on a Borgwaldt RM20H rotary smoking machine utilizing Electrostatic Precipitation (EP) to trap TPM. TPM collected in pre-weighed high purity quartz tubes was dissolved by vortexing in 30 mL tertiary butanol: water (1:1 v/v). Each 100 µL aliquot was pipetted either spiked or unspiked into a glassy carbon boat and analyzed by ETV-QQQ-ICP-MS. Mass-bias corrected ratios were utilized for data analysis and quantification of chromium. Total chromium results for the 3R4F reference cigarette (n=30 0.602 ± 0.175 ng/cig), CM7 QC cigarette (n=30 0.577 ± 0.117 ng/cigarette), and high TPM delivery commercial brand, (n=30 0.590 ± 0.085 ng/cigarette) all had TPM total chromium concentrations greater than the calculated LOD of 0.031 ng/cigarette. Though we were able to separate Cr$^{3+}$ and Cr$^{6+}$ spiked into TPM using ETV, the concentration of Cr in TPM would have to be approximately 20 times higher than observed in order to determine concentrations of the individual species.
THE EU 2015/1006 REGULATION AND ITS IMPACT ON ARSENIC FOODSTUFF SPECIATION AND QUANTIFICATION

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With the quantity of rice and rice-based foodstuffs imported into the EU growing each year, correctly distinguishing and quantifying highly toxic inorganic arsenic compounds from less harmful arsenic species that may be present in these imports has become increasingly important. In recognition of this, in June 2015 the European Union added inorganic arsenic to its regulations for setting maximum levels in a range of rice-based products. To meet the demands of the new regulation (EU 2015/1006), arsenic speciation using ICP-MS interfaced with either an ion chromatography (IC) or liquid chromatography (LC) system is required. Ion chromatography is a particularly effective technique for this analysis, because of its high selectivity and sensitive detection capability.

In addition to its speciation capabilities when coupled with IC, ICP-MS by itself is an effective tool for quantifying total arsenic concentrations in oxidized or digested food samples. By combining results from total inorganic arsenic and speciation measurements, a complete picture of the arsenic status in each sample can be generated and informed decisions about whether the inorganic arsenic component is present at levels lower than the new EU legislation can be made. In this presentation, the latest developments in arsenic speciation using IC-ICP-MS will be discussed together with an overview of total arsenic analysis using ICP-MS for a selection of food and beverage samples.
USE OF SEAWEED IN FOOD AND FEED – IMPLICATIONS FOR FOOD/FEED SAFETY

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Seaweed is the common term for marine macroalgae plants, which may be divided into green, red and brown algae types. There is an increased interest to increase the exploitation of marine macroalgae for commercial purposes including the use in relation to food and feed production. Certain seaweeds have the potential to accumulate various trace elements and contain consequently relatively high levels of both essential and toxic elements. Seaweed can even be used for bioremediation purposes in order to remove trace elements from the environment.

There is consequently a need to document the levels of toxic elements in seaweeds and to ensure that the contents do not exceed legal limits. Furthermore, a better understanding of how biological and environmental factors, like seaweed type, geography, season etc affects the levels of trace elements is called upon, in order to be able to select seaweeds with optimum characteristics for commercial use.

The present lecture will include:

1) examples of the use of seaweeds in various food items; 2) examples of the analysis of seaweed samples by (HPLC-)ICP-MS; 3) discuss the results obtained for selected toxic elements in relation to food and feed safety assessment.
DETECTION OF METAL-BASED NANOPARTICLES IN ENVIRONMENTAL AND BIOLOGICAL MATRICES USING SINGLE PARTICLE ICP-MS

Chady Stephan Ph.D., Erik Buseth, PerkinElmer, Shelton, CT

During the last decade, the production and use of engineered nanomaterials (ENMs) have experienced a drastic increase, resulting in a potential risk of their release into the environment. Therefore, the study of their impact on the environment becomes crucial. The appropriate ecological risk assessment and management of ENMs in the environment requires quantitative measurements of both exposure and effects that should, ideally, be performed by in situ analysis and give physicochemical characterization. However, most analytical techniques are not suitable for environmental matrices since nanoparticle concentrations are typically very low.

Historically, particle size has been measured by dispersive light scatter (DLS) and tunneling electron microscopy (TEM), while dissolved content has been measured by ultrafiltration. These common techniques have known limitations for measuring low concentrations in the presence of colloidal species in complex waters.

Alternatively, single particle inductively coupled plasma mass spectrometry (SP-ICP-MS) has been found to be a promising technique for detecting and characterizing metal nanoparticles at very low concentrations. SP-ICP-MS is fast and efficient and can provide more information than other currently available techniques. It can lead to the determination of particle size, size distribution, particle number concentration, and the concentration of dissolved metal. Moreover, it can distinguish between particles of different elements.

The aim of this work is to investigate the efficiency of SP-ICP-MS for the detection and characterization of metal nanoparticles in various environmental and biological matrices where they can be involved in various physicochemical processes such as dissolution and aggregation.
Influence of Rapeseed Cake Supplementation in Cow Feed on Iodine in Milk

Anicke Brandt-Kjelsen, Øyvind Enger, Giske Trøan, Egil Prestløkken and Brit Salbu

Milk is the main iodine source to the Norwegian population. The amount of iodine in iodonized salt requires us to eat 30 grams per day, which is 3 times higher than the recommended salt intake. Marine food products also contain iodine, but the majority of the population do not have a dietary intake sufficient to fulfill the recommended daily intake of 150 µg. According to the Norwegian Directorate of Health, fish accounts for approximately 20% of the population’s iodine intake, whereas dairy products account for approximately 60%. It is therefore of concern that screening of milk tanks from different regions in Norway revealed a significant reduction in the iodine concentration in milk from 2000 to 2008. Rapeseed products, more specific glucosinolates and its degradation products thiocyanates and goitrin are considered the most probable reason behind the reduced amount of iodine in milk. However, the mechanisms are not well known.

Results from our experiments confirm earlier findings that rapeseed cake significantly reduced the amount of iodine in cow milk. Correlation of plasma versus milk showed a higher transfer of iodine to milk when the cows were given soybean meal as the main protein source. The transfer was significantly reduced when soybean meal was replaced by rapeseed cake. On the other hand correlation of plasma versus urine showed a higher transfer of iodine from plasma to urine when rapeseed cake was given in place of soybean meal.

Speciation of milk using anion-exchange inductive coupled plasma mass spectrometry (AE-ICP-MS) showed that iodine was predominantly present as iodide in milk. This was especially clear at higher iodine levels (supplemented 2-5 mg/kg feed) where no difference in iodide, as % of total iodine, between the two protein sources was detected. Interestingly, at the lowest level (0.5 mg I/kg feed) preliminary results indicate that only 20% of the iodine was present as iodide when the cows were fed rapeseed cake. The majority of iodine eluted in the void, indicating that other iodine species dominate in these samples (n=5). In contrast, approximately 100% of the iodine was quantified as iodide in the samples from cows that were fed soybean meal (n=5). To our knowledge, this has not been reported earlier; hopefully I speciation analysis can provide new information regarding the mechanisms behind the reduced transfer to milk when rapeseed is added to the feed.
(O-40) IMPROVING ICP ACCURACY FOR THE ANALYSIS OF BIOETHANOL SAMPLES BY APPLYING A NEW TOTAL CONSUMPTION SAMPLE INTRODUCTION DEVICE.

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Bioethanol may contain metals and metalloids coming from either the raw material or its manufacturing, transport and/or storage. The determination of metals and metalloids in this kind of samples is difficult for several reasons: (i) the metal content is usually low (i.e., sub-ppb levels); (ii) reference materials are not commercially available; and, (iii) bioethanol samples may have a complex matrix containing up to 300 different organic compounds as well as water.

Among the possible analytical alternatives, ICP-MS appears to be an appropriate technique to carry out the quantification of metals and metalloids in bioethanol samples. This kind of determinations involves the appearance of spectral as well as non-spectral interferences, plasma thermal degradation and carbon deposits at the interface cones. A possible solution to overcome these drawbacks is to minimize the mass of sample reaching the plasma. On this subject, a dedicated low sample consumption introduction system can be employed.

In the present work, the so-called High-Temperature Torch Integrated Sample Introduction System (hTISIS) has been characterized and employed to carry out the direct analysis of bioethanol samples through ICP-MS. First, the operating conditions were optimized in order to maximize the sensitivity and simultaneously remove matrix effects. Two different flow regimes were applied: discrete and continuous. Non spectral interferences were removed and sensitivity was improved when the hTISIS was operated in both modes. This made it possible to perform analyses using 1:1 (water:ethanol) standards. Results obtained in ICP-OES were taken as reference. In order to try to validate the method, a preconcentration procedure was applied. Furthermore, real samples were spiked and the recoveries determined. The developed method was applied for the first time to the analysis of twenty-eight bioethanol real samples.

HIGH-RESOLUTION CHEMICAL PROFILING OF HETEROGENEOUS MATERIALS USING LASER ABLATION IONIZATION MASS SPECTROMETRY (LIMS)

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Sensitive and quantitative chemical insights into solid materials with high spatial resolution is of high importance in various fields of academic and industrial research, ranging from high precision analysis of materials used in the microchip industry to chemical analysis of complex structures within e.g. geological or biological samples, among many others. Accurate knowledge of e.g. contaminations or element phases within a sample material enables a better understanding of processes that may have altered the material or allows improvements of specific procedure steps in their manufacture. Measurement techniques such as Secondary Ion Mass Spectrometry (SIMS), Glow Discharge Mass Spectrometry (GD-MS), Laser Ablation Inductively Coupled Mass Spectrometry (LA-ICP-MS) are today’s state-of-the-art measurement techniques for high precision chemical analysis of solid materials with high spatial resolution. In this contribution, the measurement performance and capabilities of our Laser Ablation Ionization Mass Spectrometer (LIMS) are demonstrated presenting measurements conducted on various complex structured samples with high lateral (µm level) and vertical resolution (sub-nanometer).

The LIMS system used for measurements consists of a miniature (160 mm x Ø 60 mm) reflectron-type time-of-flight mass spectrometer allowing measurements with a high dynamic range (up to eight orders of magnitude) and detection sensitivity (10 ppb, atomic fraction). A femto-second laser system (λ = 775 nm, τ ~ 190 fs, ≤ 1mJ/pulse, laser pulse repetition rate ≤ 1kHz) is used as ablation ionization source providing e.g. increased ionization efficiencies of elements with minimized elemental fractionation effects [1]. An optical lens system focusses the laser pulses through the mass analyzer towards the sample surface to spot sizes of about 10 – 20 µm in diameter (laser irradiance at TW/cm\(^2\) level). Most recent and current measurements conducted on various sample materials, including e.g. contamination analysis of Cu electrodeposits used in the semiconductor industry [2,3], detection of fossils of micrometer dimensions embedded in an aragonite matrix [4], heterogeneous rock samples [5], among others, will be discussed to demonstrate the current measurement capabilities of our LIMS system.

References:
DEVELOPMENT OF TRIPLE QUADRUPOLE ICP-MS PERFORMANCE FOR DIFFICULT APPLICATIONS

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Several important applications in elemental analysis are difficult to address using quadrupole ICP-MS (ICP-QMS). The development of collision/reaction cells (CRC) operating in helium collision mode has improved the removal of polyatomic interferences, delivering improved accuracy for many common applications. But several problematic spectral overlaps remain, including doubly-charged interferences, direct isobaric overlaps, and very intense gas- and solvent-based polyatomics, such as O₂, N₂, CO, etc.

ICP-QMS can use reactive cell gases to resolve some analyte-interference pairs, but ion-molecule reaction chemistry does not provide a reliable solution when there are many different ions present in the cell. Quadrupole ICP-MS has no mass selection prior to the cell, so cannot prevent non-target ions from entering the cell. Existing ions may interfere with newly formed analyte product ions, or matrix components may form new product ions that overlap the masses of interest. When isotope analysis is performed using reaction mode, inter-isotope overlaps can bias the results, for example ³²S¹⁸O⁺ overlaps ³⁴S¹⁶O⁺ at mass 50.

Triple quadrupole ICP-MS (ICP-QQQ) provides a solution to these difficult interferences, using a second mass filter, positioned in front of the cell, to control of the reaction chemistry using MS/MS. With an extended mass range to allow higher-order cluster ions to be measured, a fast detector to support single nanoparticle analysis, and controlled reaction chemistry to resolve interferences including doubly-charged and isobaric overlaps, ICP-QQQ extends the range of ICP-MS analysis to new applications.

In this paper we describe examples from geochemistry, high-purity chemical analysis, environmental monitoring and the nuclear industry, to illustrate the current state of the art in ICP-MS/MS.
Short Course Abstracts

(A1)
SIGNAL-TO-NOISE ENHANCEMENT METHODS FOR ATOMIC SPECTROMETRY

Gary M. Hieftje

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Abstract

This short course is intended to provide a background in the fundamental and practical aspects of signal-to-noise enhancement, particularly as applied to atomic spectrometry. Included will be a basic discussion of the nature of signals and noise, a characterization of different kinds of noise, and how modulation can be used to distinguish between signals and noise. Several kinds of instrumental S/N enhancement will then be described and evaluated, including tuned (frequency-selective) amplification, lock-in amplification, boxcar integration, ensemble averaging, and correlation. Digital filtering and software approaches for S/N enhancement will then be compared to the hardware approaches.
(A-2) COUPLING FIELD FLOW FRACTIONATION WITH SINGLE PARTICLE ICP-MS

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(A-3) DIELECTRIC BARRIERS FOR ANALYTICAL CHEMISTRY

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Fifteen years ago the potential of a dielectric barrier discharge plasma was presented for use in analytical element spectrometry. It was a miniature planar DBD, characterized by small size, low power consumption, low gas temperature and excellent dissociation capability for molecular species. Several years later a capillary shaped DBD was presented by Na et al. applied as an efficient method for molecular mass spectrometry resulting in the development of a variety of methods now commonly termed Ambient Mass Spectrometry (AMS), which experienced a very rapid development during the last years. Within the course some techniques will be presented and tried to be characterized where dielectric barriers were used in the field of Analytical Chemistry.
A4

BASIC SEPARATION METHODS FOR SPECIATION ANALYSIS

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The course is an introduction for beginners in speciation analysis and describes:
Basic theory of high performance liquid chromatography (HPLC) including system suitability parameters as efficiency and resolution
Chromatographic separation systems including normal phase, reversed phase, ion exchange, ion-pair and size exclusion chromatography and their individual advantages and limitations
The practical hyphenation of the separation system to the ICP-MS
Validation of an LC-IC-MS method including linearity, precision, accuracy, limits of detection and determination and column recovery
(A5)
LASER ABLATION ICP-MS WITH LIBS AND LAMIS

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Applied Spectra, Inc, Fremont, CA USA,
(B1)
PLASMA-SOURCE TIME-OF-FLIGHT AND DISTANCE-OF-FLIGHT MASS SPECTROMETRY – PART 1

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ANALYSIS OF ENERGY STORAGE MATERIALS: RECENT TRENDS AND APPLICATIONS

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Lithium-ion batteries are widely used in modern consumer electronics and are increasingly used for electric vehicles. Due to the high energy density and good cycling stability these energy storage systems become more and more important for the economy. The main disadvantage of the batteries is limited cycle life due to unwanted chemical and interfacial reactions for example of the electrodes and electrodes. Since composition of lithium ion batteries is complex and challenging in terms of chemical analysis (inorganic fluorinated salt, organic solvents, transition metals oxide/ carbon based electrodes and influence of temperature and electrochemical energy), investigations cannot just start be carried out by one method to gain access to all phenomena. To gain a deeper knowledge about the fundamental decomposition processes, novel techniques and methods have to be developed and systematic research of the degradation reactions has to be performed.

Until today, numerous attempts on characterization of these reactions and on elucidation of the aging mechanisms were performed. However, there is only a limited number of approaches trying to clarify the mechanisms. To the present day, fundamental understanding of the aging mechanisms is not achieved.

This short course will focus on the usage of ICP based methods in the field of lithium ion battery research. A brief overview about routine application will be presented as well as recent method development and application to the current challenges.
(B3)
APPROACHES TO EFFICIENTLY INTRODUCE LIQUID SAMPLES INTO ICP INSTRUMENTS

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An accurate and reliable analysis through Inductively Coupled Plasma may depend on the proper sample introduction into the excitation or ionization cell. In the case of liquid samples, the key processes are the aerosol generation and the droplet size selection. For many years these steps have been considered as critical for elemental analyses. The research efforts done on this subject have led to new and more appropriate ways for liquid sample introduction. The present short course gives an overview of the fundamentals of these two steps as well as recent advances in the field of solutions introduction into ICP. New fundamental researches have made it possible to develop efficient liquid sample introduction systems. Thus, for instance, studies carried out with currently employed pneumatic nebulizers demonstrate that the mechanism of droplet production may be severely influenced by slight changes in the nozzle configuration. Once the aerosol is generated, relevant variables affect the way in which it is transported towards the plasma. Among them, temperature has revealed to be extremely important. Besides, the performance of systems recently developed will be discussed taking conventional devices as reference. The comments will be focused on: (i) multifunction systems, able to simultaneously work with liquid and vapor generation; (ii) devices that allow on-line application of standard additions; (ii) low sample consumption devices, are able to increase the aerosol fineness and the transport efficiency; and, (iii) desolvation systems. Advices will be given to improve the performance of the spectrometer by acting on the sample introduction system and selecting the most appropriate approach for a particular application.
(B4) SPECIATION ANALYSIS – WHY, WHEN AND HOW?

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This short course will introduce the novice into the aim, concept and methodology of speciation analysis. The necessity of obtaining relevant information about the consequences of the presence of chemical species in the environment, biological organisms or simply materials is the driving force for performing speciation analysis. We will then discuss the tools available for speciation analysis with a focus but not restricted on those related to plasma spectrometry. How to obtain accurate results and to avoid artifacts during the analysis will be also discussed in some detail.
PLASMA-BASED SOURCES FOR DIRECT ANALYSIS: FUNDAMENTALS AND APPLICATIONS OF AMBIENT DESORPTION/IONIZATION MASS SPECTROMETRY

Carsten Engelhard$^1$ and Jacob T. Shelley$^2$

Presented by Carsten Engelhard

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The ultimate goal of analytical chemistry is to provide, what G. E. F. Lundell described as, “the chemical analysis of things as they are” such that a comprehensive assessment of sample constituents is directly obtained in a way that is nondestructive, while the sample is interrogated in its native environment. Recent efforts in mass spectrometry ionization source development have demonstrated these attributes to be possible. In such ambient mass spectrometry experiments, the source desorbs molecules from a surface, softly ionizes them, and transfers these ions into a mass spectrometer. This course will cover fundamental principles of desorption/ionization processes, as well as applications of these ionization sources. A particular emphasis will be placed on plasma-based systems.
“FUNDAMENTALS AND APPLICATIONS OF DEPTH PROFILE ANALYSIS BY GLOW DISCHARGE SPECTROMETRIES FOR BEGINNERS”

Rosario Pereiro

Glow discharge coupled to spectrometric techniques (e.g. optical emission and mass spectrometry) has become a versatile tool for depth profile elemental analysis of layers (with thicknesses that can range from a few nanometers to several microns) and interfaces. The almost independence of sputtering and excitation/ionization processes makes simpler the development of quantitative approaches for quantitative depth profiling (concentration versus depth) as compared to other direct solid analysis techniques.

In this course, an introduction to multimatrix quantification fundamentals for depth profile analysis by GD coupled to optical emission and mass spectrometry will be presented. Moreover, examples of practical interest in varied fields such as thin film solar cells, galvanized materials, coated glasses, ceramics, etc, will be given.
(C3)
HOW TO COLLECT RELEVANT AMBIENT AND OCCUPATIONAL AIR SAMPLES FOR PLASMA BASED SPECTROCHEMICAL ANALYSIS

Davy Rousset

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Because of analytical progress these last decades, plasma-based spectrochemical methods can now reach ranges of concentrations and achieve levels of precision and reproducibility which cover most of the needs in term of quantification of element or chemical species in their environment.

The main source of uncertainty in this determination results in the significance of the collected sample with regard to its environment and, in lesser extent, the preparation of the sample for analysis. In the case of air sampling, the sampling strategy plays a major role in this uncertainty because it has to take into account both the spatial and temporal variability of the chemical agent concentration in the air, as well as the objective of the measurement itself. For environmental health issues, the objective of measurement is to determine levels of concentration of chemical agents in ambient air to which the whole population may be exposed. In industrial hygiene, this objective is mainly the occupational exposure assessment to chemical agents by sampling and analysis of workplace air in the immediate vicinity of workers. This difference is fundamental and deeply influences sampling and associated analytical methods to assess concentrations of chemical agents in the air.

This short course will then illustrate with concrete examples the resulting differences between occupational and environmental health issues for the following steps in exposure assessment:

Definition of the conventional size-fractions;
Personal/static air samplers (efficiency, bias);
Working ranges of concentrations (flow rates, sampling durations);
Sample Preparation (sampling medium, digestion methods for refractory elements, direct analysis, sampler wall deposits);
Analysis.
(C4)
SOURCES OF CONTAMINATION AND REMEDIAL STRATEGIES

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In theory, state of the art inductively coupled plasma mass spectrometry (ICP-MS) instrumentation possesses the prerequisite sensitivity to carry out multi-elemental trace analyses at sub-ng l\textsuperscript{-1} to sub-pg l\textsuperscript{-1} levels in solution. In practice, constraints mainly imposed by various sources of contamination in the laboratory and the instrument itself, as well as the need to dilute sample solutions prior to analysis ultimately limit the detection capabilities. Here we review these sources of contamination and wherever possible propose remedial strategies that we have found efficacious for ameliorating their impact on the results of multi-elemental trace analyses by ICP-MS.
INTRODUCTION TO MASS CYTOMETRY

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Abstract

Mass cytometry is a multiparameter analysis technique based on detection of metal-conjugated antibodies in single cells via inductively coupled plasma time-of-flight mass spectrometry.

The short course will focus on the fundamentals of the cell introduction system, ion generation, transport and mass analysis and data handling and processing. Use of specially designed elementally embedded polystyrene beads as calibration standards for single-particle detection will be discussed. Cell sample preparation aspects of surface and intracellular staining, nucleic acid 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. Recent developments in the area of laser-ablation-based imaging of immunostained tissues and cells will be discussed.
ANALYTICAL STRATEGIES FOR METALLOPROTEINS ANALYSIS

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The course will describe metallomics approaches to the importance of finding and quantifying metalloproteins and selenoproteins. The different steps, i.e sample preparation, separation, detection, data processing, of mass spectrometric metallomics analysis will be detailed. The covalent and non-covalent aspect of the binding between the heteroelement and the protein will be underlined. In terms of separation techniques, mostly liquid chromatography and gel electrophoresis will be featured. The detection methods will focus on ICP MS to seek for heteroelement-containing proteins and when possible to reduce the complexity of the ESI MS mass spectrum search. Samples from different origins (human, toxicology, ecotoxicology) will be illustrated as metallomics applications.
(D3) MATRIX EFFECTS IN ICP TECHNIQUES

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Matrix effects can be considered as the most important drawback of Inductively Coupled Plasma techniques. Although these methods were initially considered as to be free from interferences, it has been subsequently verified that the main sample component has a direct influence on the accuracy of the determinations. A classification of matrices can be established considering acids, organic solvents and dissolved inorganic salts. In order to overcome matrix effects it is necessary to understand the origin of these unwanted effects. Each one of the mentioned concomitant has its own effects on the performance of the system. Thus, organic solvents modify the aerosol generation, its transport efficiency and the plasma thermal characteristics. Meanwhile, acids and inorganic salts have a noticeable influence on the aerosol transport and the analyte atomization/ionization/excitation, respectively. Furthermore, when considering ICP-MS all these species cause spectral interferences.

The present course deals with the detection of the source of the ICP-OES and ICP-MS interferences and the ways to overcome them. Results based on selected applications (clinical, fuels, environmental analysis…) will be discussed.
(D4)
PLASMA-SOURCE TIME-OF-FLIGHT AND DISTANCE-OF-FLIGHT MASS SPECTROMETRY - PART 2

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Elemental chromium, trivalent chromium \([\text{Cr(III)}]\) and hexavalent chromium \([\text{Cr(VI)}]\) are commonly observed chromium species in the environment and are known to interconvert. \text{Cr(VI)} is highly toxic and it is known to be a carcinogen. Therefore, SI traceable procedures for the accurate quantification of \text{Cr(VI)} in the presence of the other Cr species are urgently required since they will be invaluable for quality control of higher throughput tests by field laboratories. An HPLC-ICP-MS method was developed, achieving excellent retention and separation of inorganic Cr species within 15 min, (see figure 1A). By diluting the samples before analysis, samples from the Aquacheck Proficiency Testing scheme [1] were measured with the developed method. Chromium species data obtained by double spiking single IDMS calibration on the same samples (to account for possible species redox interconversion) agreed well with those obtained using external calibration. [2]. The developed measurement capability was demonstrated through participation in an international inter-laboratory comparison study (CCQM K124 Part B) which was organised by the Government Laboratory of Hong Kong. For this study, spiking, dilution (1:6) and stabilisation were achieved in one step by the gravimetric addition of an appropriate amount of \(^{53}\text{Cr(VI)}\) in diluent solvent to the sample [Cr (VI)-spiked water] to achieve a target isotope ratio of 1:1 for \(^{52}\text{Cr(VI)} : ^{53}\text{Cr(VI)}\). A relative expanded uncertainty of 1.42\% (k=2) for a level of \text{Cr(VI)} of 62.88±0.90 µg kg\(^{-1}\) was achieved. The value reported by LGC (see figure 1B) is in good agreement with the consensus mean therefore the procedure is suitable for high accuracy measurement and for the certification of reference materials.

**Figure: 1A** Example chromatogram of CCQM K124 Part B blend ≈10 ng g\(^{-1}\)  
**Figure: 1B** Participant results for CCQM K124 Part B (mean & ±1SD)

[1] LGC proficiency testing scheme for water testing laboratories- Aquacheck.  
DETERMINATION OF METHYLMERCURY AND INORGANIC MERCURY SPECIES IN SEAFOOD BY HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC) COUPLED TO AN ICP-MS

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Mercury occurs naturally in the environment in elemental, inorganic and organic (methylated) forms. Methylmercury is the dominant form of mercury found in aquatic animals, including fish. Marine seafood is one of the major sources of mercury in the human food chain. Cooking and heat do not reduce the mercury levels.

A method is developed for the simultaneous determination of methylmercury (MeHg⁺) and inorganic mercury (iHg) species in seafood samples. The study focuses on the extraction and quantification of MeHg⁺ (the most toxic species) by HPLC and detection by ICP MS at mass-to-charge ratio 202.

The following instrumentation was used: Varian 920 LC Liquid Chromatography station and Varian 820-MS Inductively Coupled Plasma Mass Spectrometric detector.

Extraction efficiency was estimated by comparing the results of measurements of total Hg by ICP-MS with the sum of methyl and inorganic Hg determined in extracts by HPLC/ICP-MS. The spikes were used for HPLC/ICP-MS measurements quality control.

Mercury (Hg) compounds were extracted from 0.3 - 0.5 g edible seafood or 0.1 - 0.3 g lyophilized seafood material by adding 10 ml – 50 ml aqueous 1% - 5% w/v l-cysteine· HCl·H₂O and heating 60 – 120 min at 60 – 80 °C in glass vials.

Hg compounds in 20 μl of filtered extract were separated by reversed-phase High Performance Liquid Chromatography using a C-18 column at room temperature.

Recovery of added analyte into lyophilized squid was 77% - 81% for methylmercury and 92% - 104% for inorganic Hg. But recovery of added analyte into lyophilized fish was 82% - 88% for methylmercury and 84% - 94% for inorganic Hg.

The repeatability relative standard deviation (RSD r) ranged from 2.7 to 6.8 % for inorganic Hg and from 2.5 to 7.6 % for methylmercury while the reproducibility relative standard deviation (RSD R) ranged from 4.7 to 10.2 % for inorganic Hg and from 2.9 to 9.1 % for methylmercury. The method proves to have acceptable precision for test materials, thus it can be used for its intended analytical purpose.
DETERMINATION OF PALLADIUM IN ENVIRONMENTAL SAMPLES BY ICP-MS TECHNIQUES

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It is well known that platinum group elements (PGEs) are emitted to the environment from the catalytic converters of automobiles. ICP-MS technique has commonly been used to determine low PGE mass fractions in environmental samples for monitoring purposes. However, spectral interferences cause challenges especially in the determinations of Pd. The list of potential interferents in the determinations of Pd on m/z 105, 106 and 108 includes many isobaric and polyatomic species, such as cadmium (Cd\textsuperscript{+}), and various oxides (YO\textsuperscript{+}, ZrO\textsuperscript{+}, MoO\textsuperscript{+}), hydroxides (SrOH\textsuperscript{+}), argides (CuAr\textsuperscript{+}, ZnAr\textsuperscript{+}) and chlorides (ZnCl\textsuperscript{+}). Resolving all of these spectral interferences has not been possible even by using high resolution ICP-SFMS. Thus, to achieve reliable results, chemical separation methods (e.g., ion exchange, extraction methods or co-precipitation) are often employed prior to ICP-MS determinations. Furthermore, desolvating sample introduction systems or collision/reaction gases have been used to minimize the interferences.

In this work, two approaches were used to eliminate the interferences in the determination of Pd mass fractions in microwave-digested road dust and moss samples. The more traditional approach combined a cloud point extraction method with ICP-QMS determination, whereas the other approach utilized the novel ICP-MS/MS technique with a collision/reaction cell in the elimination of spectral interferences. The results obtained using these methods were in a good agreement with each other. However, the need for sample pretreatment was significantly reduced using the ICP-MS/MS approach.
COMPARISON OF THE INFLUENCES OF DIFFERENT LITHIUM ION BATTERY ADDITIVES ON THE ELECTROLYTE AGING BY SIMULTANEOUS ONLINE COUPLING OF TWO DIMENSIONAL ION CHROMATOGRAPHY TO INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY AND ELECTROSPRAY IONIZATION MASS SPECTROMETRY

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The Lithium-Ion Battery (LIB) plays an important role in portable electronic devices such as mobile phones or laptop computers because of its high energy density. Still the LIB has a limited lifetime due to capacity fading. The fading is caused by aging processes of the battery components. Concerning the LIB electrolyte the aging is caused by reactions of the conducting salt lithium hexafluorophosphate (LiPF₆) with the electrolyte solvents. One group of decomposition products which is formed during aging are alkyl phosphates. They have been identified by ion chromatography (IC) coupled to electrospray ionization mass spectrometry (ESI-MS). The quantification of these alkyl phosphates can give information about the kinetics of the decomposition. Furthermore it is possible to use the alkyl phosphates as markers for the degradation of LiPF₆. The quantification of the alkyl phosphates is complicated because it is not possible in either IC or ESI-MS. With IC they cannot be detected separately from fluoride by conductivity detection because of co-elution. By using ESI-MS they can be detected, but not quantified due to the non-availability of standard materials. Therefore the quantification with ICP-MS using a structurally similar phosphorus containing standard is the method of choice. Due to the selectivity for phosphorus the detection is not influenced by fluorine. A two-dimensional chromatography further allows a separation from other fluorophosphates and to separate the alkyl phosphate species on the second column.

During this work the alkyl phosphate species in thermally aged electrolyte were quantified in different samples. Their amount was found to be in the lower range of mmol/L. In order to enhance the performance and safety of the LIB, different additives are added to the electrolyte. The influence of these additives on the alkyl phosphate formation has been investigated and major differences to the aging behavior without additives have been detected.

Literature:
(P-5)
DEVELOPMENT OF ANALYTICAL STRATEGIES TO STUDY SELENOPROTEIN
EXPRESSION IN HUMAN CELL LINES

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Selenium (Se) is an essential trace element, which is incorporated as a rare aminoacid, selenocysteine, in
twenty five selenoproteins, to constitute the selenoproteome. Selenoprotein family is one of the most
important bioactive form of selenium in human health. Initially demonstrated in Kashin Beck and Keshan
diseases, selenium deficiency is associated with several pathological conditions, including cancer,
neurodegenerative diseases, immune and muscular disorders. Chronic selenium deficiency is
hypothesized to decrease antioxidant defenses and redox regulatory pathways through a dysregulation of
selenoprotein expression. We are interested in understanding the synthesis and regulation of human
selenoproteins, which is critically dependent on the availability of adequate analytical methodology.

To understand the function and regulation of human selenoproteome, which is expressed at a trace levels,
it appears critical to develop innovative strategies based on a multidisciplinary approach to detect and
quantify selenium by various elemental and molecular mass spectrometer tools. First, selenium has a
particular isotopic profile with six stable isotope ($^{74}\text{Se}$, $^{75}\text{Se}$, $^{77}\text{Se}$, $^{78}\text{Se}$, $^{80}\text{Se}$ and $^{82}\text{Se}$) used as a signature
in our analysis with ICP-MS or ESI-MS/MS. In parallel, the use of isotopically enriched selenium also
allows cellular labelling and tracing of selenoproteins and other seleno-couponds. By coupling liquid
phase separation methods (HPLC) with specific mass spectrometry analytical tools, we have developed
several methods for detecting several selenoproteins simultaneously in various human cell lines.
SPATIALLY RESOLVED ANALYSIS OF THE LITHIUM DISTRIBUTION ON CYCLED ELECTRODES BY LASER ABLATION – INDUCTIVELY COUPLED PLASMA - OPTICAL EMISSION SPECTROMETRY (LA-ICP-OES)

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Being successfully introduced into the market, lithium-ion batteries (LiBs) are already state-of-the-art for portable electronic devices and one of the most promising candidates for high capacity energy storage. Besides relatively high manufacturing costs and a long charging time, the major drawback is the degradation of the cell components, which is called aging and which minimizes both storage lifetime and cycle lifetime.

With its very complex system of electrodes, separator and electrolyte, the spatial lithium distribution on electrodes could not be investigated and elucidated with just one method. Inductively coupled plasma - optical emission spectrometry (ICP-OES) coupled with laser ablation (LA) is an attractive combination for the spatially resolved investigation of lithium.

The focus of this work is the spatially resolved analysis of the lithium distribution on cycled anodes (mesocarbon microbeads, MCMB) in self-assembled pouch bag cells. The test cells were cycled for 100 times state of charge (SOC) of 0%. After the aging process, the cells were disassembled in an argon-filled glove box to prevent contact with air. The MCMB electrodes were rinsed with an organic carbonate to clean it from leftovers of the electrolyte and afterwards were dried in an argon atmosphere. Subsequently, the spatial lithium distribution was investigated by means of LA-ICP-OES. The spatial resolved analysis of lithium is important to confirm the assumption of dendrite growth, which leads to short-circuits, and to locate the region of lithium distribution on the anode, which affects the performance of LIBs.
ADAPTATION OF GLOW-DISCHARGE SECTOR FIELD MASS SPECTROMETRY IN THE FIELD OF BATTERY RESEARCH

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The lithium ion battery (LIB) technology, introduced in the early 90’s, opened a whole new market for high power and high energy portable devices. Recent applications in the electromotive industry as well as in the field of stationary storage systems for a more efficient use of renewable energies attracted the LIB technology [1].

The state of the art lithium ion battery consist out of graphite, with a decent capacity of 372 mAh g⁻¹, and mostly layered lithium metal oxide (LiMO₂) cathodes, supplying around 150 mAh g⁻¹ [1]. One of the most interesting layered lithium metal oxides is LiNi₁/₃Co₁/₃Mn₁/₃O₂ (NCM 111) as cathode material which show several desired properties such as high capacity, less toxicity, lower cost and milder thermal instability in the charged state. Nevertheless, depending on the application LIBs still show severe drawbacks, especially the limited storage lifetime (= calendar life) and cycling lifetime (= cycle life). These criteria are closely related to the degradation of the battery cell components, which is known as cell aging. The dissolution of transition metals from the cathode is proposed to have a major influence on the cycle life due to the deposition of those metals on the graphitic anode. It has been shown for lithium manganese oxide (LiMn₂O₄) spinel type cathodes that the deposition of manganese leads to cracking and contamination of the solid electrolyte interphase (SEI) and thus to ongoing lithium consumption. [2]

The failure mechanisms depend on the cathode material of which the LiMn₂O₄ spinel type electrode degradation mechanism is well known, where a disproportionation (2 Mn³⁺ → Mn⁴⁺ + Mn²⁺) and acid dissolution by HF are assumed [2]. In here we analyze the degradation products of the layered lithium metal oxide Li₁Ni₁/₃Co₁/₃Mn₁/₃O₂ (NCM) and the influence of the transition metals on the SEI.

To understand the influence of transition metal dissolution of the NCM cathode on the protecting SEI surface, GD-MS is an excellent tool in the detection of deposited elements. Furthermore, with pulsed operated GD sources it is possible to obtain excellent spatially resolved information with respect to the depth resolution. In here, we use the Glow Discharge Mass Spectrometry to quantify the deposition of transition metals deposited onto the graphitic anode as well as the contents of lithium. We prepared matrix matched standards with elevated contents of Li, Ni, Co and Mn in order to perform external calibration. The bulk concentrations of the spiked elements are validated via ICP-OES. Afterwards, the calibration is adapted on cycled cells in order to get information on the contents of the elements especially in depth.


SYNTHESIS AND APPLICATION OF A MATRIX MATCHED EXTERNAL STANDARD MATERIAL TO QUANTIFY MANGANESE IN LITHIUM ION BATTERY ELECTRODES BY MEANS OF LASER ABLATION INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY

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Due to a lack of reliable methods for μm scale determination of the elemental distribution in aged lithium ion electrodes, we developed a method using laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS). The solid electrode sample was investigated using a 193 nm ArF Excimer Laser with spot sizes ranging from 2.5-150 μm and subsequently analyzed in an ICP-MS. Due to the evaporation of small parts of the sample with the laser and analyzing the elemental composition of each spot allowed a three-dimensional investigation of the electrode composition.

Considering the cathode/anode active material combination of LiNi\(_{1/3}\)Co\(_{1/3}\)Mn\(_{1/3}\)O\(_2\) (NMC) and graphite as a standardized and well-known setup for lithium ion batteries (LIBs) [1], it shows performance loss over time. Various causes, such as capacity decrease or power fading have been proposed or identified [2]. One major factor concerning the performance of this system is the unwanted manganese dissolution followed by the deposition on the anode.

The synthesized NMC/graphite composite allows us to quantitatively study the manganese content in a single digit μm-range on the surface and inside the bulk material of the anode.

INVESTIGATION OF LITHIUM-ION BATTERY ELECTROLyTES BY A GAS CHROMATOGRAPHY - BARRIER IONIZATION DISCHARGE DETECTOR

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In recent years, lifetime and safety of lithium-ion batteries (LIBs) became more and more important for automotive applications such as electric or hybrid electric vehicles.[1] Most commercially available electrolytes in LIBs contain lithium hexafluoro-phosphate (LiPF₆) as conducting salt and organic carbonates, e.g. diethyl carbonate (DEC) and ethylene carbonate (EC) as solvents.[2] These organic carbonates are necessary to provide sufficient solubility of LiPF₆, high ion mobility and a wide working temperature range. During cycling these compounds can decompose on many different ways and this is the main reason for the limited lifetime of LIBs. A selection of possible decomposition products resulting from thermal or electrochemical decomposition are polymers, transesterification products, fluorinated phosphates, alkylphosphates or CO₂. Several gas chromatographic (GC) methods were developed in the last years to investigate the volatile decomposition products of the LIB electrolytes.

During this work, the volatile decomposition products were investigated by GC - barrier ionization discharge (BID). The BID is a relatively new universally usable detector compared to the flame ionization detector (FID). It works with a 17.7 eV helium plasma that ionizes almost all compounds except neon and the plasma gas (He) is not detectable. The sensitivity is more than two times higher compared to FIDs and over 100 times higher than thermal conductivity detectors (TCDs).[3] Therefore, standard electrolytes, electrolyte from self-built LIBs and gas samples were investigated and compared to the results of GC-MS and GC-FID.

References:
(P-10) QUANTIFICATION OF ORGANOPHOSPHATES AS AGING PRODUCTS OF LITHIUM-ION BATTERY ELECTROLYTES BY MEANS OF GAS CHROMATOGRAPHY - INDUCTIVELY COUPLED PLASMA SECTOR FIELD MASS SPECTROMETRY (GC-ICP-SF-MS)

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Commercial electrolytes of lithium-ion batteries have to meet challenging requirements regarding performance, cycling stability and safety. Lithium hexafluorophosphate (LiPF₆) as conductive salt in combination with organic carbonates as solvents is used as the state-of-the-art electrolyte. Due to high hygroscopic properties of LiPF₆, the electrolyte contains water impurities leading to hydrolysis of the conducting salt which, eventually, causes failure of the battery by forming gaseous PF₅ and subsequently hydrofluoric acid. Furthermore, this decomposition of the conductive salt in the presence of water leads to the formation of PO₂F₃. This very reactive species that can interact with the carbonates in the electrolyte and form a variety of organophosphates (Figure 1).[1]

![Reaction Scheme](image)

Figure 2: Proposed decomposition route of the hydrolysis of lithium hexafluorophosphate in an organic carbonate based electrolyte; as a comparison the molecular structure of the chemical warfare agents sarin and soman is given.

The qualification and, even more important, the quantification of organophosphates is of special interest due to the structural similarity with chemical warfare agents (CWA) like sarin and soman (Figure 1) and therefore high potential toxicity.[2] Since no molecular standards are commercially available for the analytes, the demand of quantification was met by using an inductively coupled plasma in combination with a sector field mass spectrometer (ICP-SF-MS) as detection method; after separation with a gas chromatograph (GC) and introduction of a liquid internal standard into the plasma; a quantification of the respective substances could be obtained. The high resolution of SF-MS allows a detection of the phosphorus signal in medium resolution (4000) sufficient to avoid atmospheric polyatomic interferences like ¹⁴N¹⁶O¹H or ¹⁵N¹⁶O⁺. The identification of the analytes was performed with a novel Q Exactive™ GC Orbitrap™ GC-MS/MS device; with this high resolution setup, the chemical structures of organophosphates were proposed by their exact masses.

(P-11)

NANO-PARTICLE ANALYSIS USING DATA ACQUISITION DWELL TIMES BETWEEN 10µS AND 50µS WITH A DYNAMIC RANGE EQUIVALENT TO MORE THAN 1E9 CPS

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Inductively coupled plasma mass spectrometry (ICP-MS) is proving to be a useful tool for characterization and quantification of metallic nanoparticles. However, the short (150 – 400 µs) transient signals generated by single particle events present challenges to current ICP-MS instrumentation. Minimum dwell times are available now in the 50-100µs timings but limitations in sensitivity for quadrupole ICP-MS make analysis of smaller particles difficult below 50µs. This work will present data acquired with dwell times down to 10µs for a range of nanoparticles. The advantages of the higher sensitivities possible with the Nu AttoM will be demonstrated. We will describe the unique detection system of the AttoM which can measure fast transient signals to >e\textsuperscript{7} cps. We will also describe an additional range extension facility based on physical attenuation of the ion beam which allows the same faster dwell times to be used for signals in excess of 1e\textsuperscript{9} cps. High signal to noise detection of 10nm particles will be demonstrated along with the ability to display and process nanoparticle data within the Nu Quant data processing package. The flexible capabilities of NICE scripts (Nu Instrument Calculations Editor) to customise the processing and reporting of particle numbers and sizes will be shown with the charting capability to report multiple distributions from a single data collection. The data processing methods allow easy discrimination of 15nm and 20nm particles from significant levels of ionic background signals with graphical and tabulated reports available for particle size, particle concentration, ionic concentration, detection limits, calibrations and size distributions with normal and log-normal fitting.

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{histogram.png}
\caption{Histogram of particle sizes Au15 nm 30 nm 40 nm 2}
\end{figure}
(P-12)
3D MODELLING OF PLASMA FLOW AND SPATIAL DISTRIBUTION OF TEMPERATURE IN THE PLASMACHEMICAL REACTOR


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Nonstationary three-dimensional gas flow dynamics and spatial distributions of pressure and temperature in the plasma torch were calculated using four-dimensional model of the plasma. Quartz torch by Steco LTD (Saint-Petersburg, Russia) was used as an object of investigation with radio frequency generator VCHI11-60/1.76 and condensation chamber.

As a result of modelling temporal evolution of plasma flow velocities, pressure and temperature in the torch and chamber were obtained. Such effects as reverse flow, pulsation and rotation of the plasma, circulating precursor flow near the swirl were discovered and experimentally verified. It is found that the geometry of the torch is not optimal and causes reverse flow that lowers efficiency of the synthesis of nano- and microparticles. Different variants of the torch were tested numerically. Optimal geometry of the torch, chamber and gas flows structure were found.

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(P-13)
3D MODELLING AND OPTICAL DIAGNOSTICS OF GAS FLOW DYNAMICS NEAR THE ICP-MS INTERFACE


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Thanks to combination of unique parameters (record sensitivity and dynamic range, possibility of multielemental and isotopic analysis) inductively couple plasma mass spectrometry (ICP-MS) is one of the most effective methods of elemental analysis.
One of the key peculiarities of ICP-MS spectrometer is necessity of transition from atmospheric pressure where the torch operates to high vacuum for ion detection. The interface system serves for this reason. It also plays significant role in formation of ion flux, thus, determines analytical characteristics of spectrometer. Inner space of interface is covered in serial spectrometers, so for investigations a prototype Elan DRC-II (PerkinElmer, USA) was built.
Three-dimensional model of the “inductively coupled plasma – mass spectrometer interface” was created. Nonstationary gas flow dynamics and spatial distributions of pressure at the torch outlet, near the sampler cone orifice and inside the interface were calculated. Tangential part of plasma velocity and its rotation in front of the interface lead to ion flux oscillations inside the interface. Calculated results are in good agreement with schlieren visualization.

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(P-14)
STRATEGIES FOR DEALING WITH HIGH MATRIX SAMPLES USING ICP-MS

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Elemental analysis in high matrix samples can be a challenge in ICP-MS. Matrix effects include suppression of the internal standards, signal drift and deposition on the interface cones of the instrument. The most common way to overcome high matrix load issues is to dilute the sample to reduce the amount of matrix entering the instrument. Dilution of the sample obviously also leads to a dilution of the analytes present, so the dilution factor needs to be chosen carefully in order to maintain the detection limit performance required.

While routine methods such as EPA method 200.8 (Drinking Water) or EPA method 6020 (Waste Water) are performed in many laboratories, skilled technicians are still required to set-up and prepare the analysis, as well as to actively monitor the results and perform further sample manipulation as required throughout the analytical run. Measurements which have not passed certain criteria, such as internal standard recovery, analyte concentration range or stability would need to be repeated manually. A combination of ICP-MS together with an intelligent and flexible, automated dilution system is therefore ideal for analysing heavy matrix samples, especially for high-throughput laboratories.

In this presentation, results from the fully automated, high-throughput analysis of drinking waters, in accordance with U.S. EPA Method 200.8 using ICP-MS coupled with an autodilution system will be presented. In addition, data for concentrated NaCl solutions will be shown to demonstrate the performance and flexibility of the system for handling very high and variable matrix samples.
(P-15)
EXPANDING THE DETECTABLE NANOPARTICLE SIZE RANGE BY SECTOR FIELD ICP-MS

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There is ever increasing interest in analyzing smaller nanoparticles by single particle ICP-MS. This is however, hampered by the sensitivity and background of most instrumentation. Due to the high ion transmission and the low dark noise of Sector Field ICP-MS, reliable detection and size estimation of single digit nanoparticles is presented.
USE OF $10^{13}$ OHM FARADAY CUP AMPLIFIERS IN (LA-) MC-ICP-MS: EXTERNAL PRECISION OF $^{234}\text{U}/^{238}\text{U}$ RATIOS AND $\Delta^{11}\text{B}$

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High-precision isotope ratio measurements are made possible by using mass spectrometers with arrays of Faraday cups, since this detector type is highly stable and linear over a wide dynamic range of ion beam intensities. However, for samples where the total analyte quantity is limited or where low-abundance isotopes are measured, measurement precisions for the resulting low intensity ion beams are limited by electronic baseline noise. Thermo Scientific $10^{13}$ ohm Faraday cup current amplifiers improve the signal/noise ratio. External reproducibility of isotope ratio measurements from ion beams with intensities from 3 - 200 fA (0.3 – 20 mV on standard $10^{11}$ Ω amplifier, or ca. 20 kcps – 1.25 Mcps) intensity are significantly improved when compared against those measured with standard amplifiers [1]. New proprietary technology means that the settling times of the $10^{13}$ ohm amplifiers are not greatly extended. Thus, with care for signal stability, application in laser ablation (LA-) MC-ICP-MS could be feasible, improving precision for high-spatial resolution isotopic measurements.

Data from a Thermo Scientific NEPTUNE Plus MC-ICP-MS with Jet Interface option for highest-sensitivity, demonstrates exceptional mass bias and detector stability. The reproducibility of $^{234}\text{U}/^{238}\text{U}$ ratios with 1 pg $^{234}\text{U}$ consumed per run was 0.6 ‰ (2σ) using a Faraday cup with $10^{13}$ Ω current amplifier for the $^{234}\text{U}$ beam.

At the other end of the mass spectrum, availability of B from marine carbonate samples can limit precision. We present new $\delta^{11}\text{B}$ data from both solution mode and laser ablation mode using $10^{13}$ ohm amplifiers. The external reproducibility of solution mode data are compared to data measured by standard amplifiers; LA data against multi-ion counter (MIC) data.

INTERNAL STANDARDIZATION IN ICP-MS USING A DUAL MODE SAMPLE INTRODUCTION SYSTEM

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Internal standardization is an efficient way to correct instrumental drift and matrix effects during inductively coupled plasma mass spectrometry (ICP-MS) measurements. The internal standards should be carefully chosen to have same kind of characteristics than the analyte, particularly mass number and ionization potential. In addition the samples should not contain measurable amounts of the chosen internal standard and the intensity changes of the internal standard should be proportional to that of the analyte.

Hydride generation (HG) is widely used to improve sensitivity when measuring hydride forming elements, for example arsenic. The elements are reduced typically with sodium borohydride in acidic media to form gaseous hydrides which are introduced to the plasma. The analyte is separated from the matrix in a gas liquid separator and the sample introduction efficiency increases significantly to nearly 100%. Internal standardization can be used also with hydride generation to correct instrumental drift (and possibly some deviation caused by the sample introduction system). With the conventional HG-ICP-MS setup the internal standards have to be hydride forming elements and there are only a few options of which to choose.

In this study a dual mode sample introduction system (Thermo Elemental) was used to combine vapor generation and liquid nebulization in sample introduction to the ICP source. The used torch had two inlets and the separate sample gas flows were mixed before the plasma. With this arrangement it was possible to measure both liquid and hydride forming internal standards during the same measurements. Ge and Bi were introduced to the plasma as hydrides and Y and Pd were introduced as a liquid aerosol. Arsenic was measured by dual mode HG-ICP-MS and the correction abilities of the different internal standards were compared.
THE DETERMINATION OF MERCURY IN PLASTICS BY COLD-VAPOR-GENERATION (CVG) INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETRY (ICP-OES).

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Determination of mercury in different matrices is an important task due to the health effects related to the Hg. Special attention should be paid to the concentration of mercury in microplastics in aquatic or in marine environments because marine organisms are able to accumulate Hg. Recent research show that plastics in marine environment can be ingested by different organisms and therefore microplastics present potential risk of enhancing Hg accumulation in food chain.

In this work cold vapor generation technique with ICP-OES (PerkinElmer Optima 8300) was used in the analysis of mercury in certified polyethylene (PE), polypropylene (PP), polyvinyl chloride (PVC) and acrylonitrilebutadienestyrene (ABS) samples. Samples of 150 mg were digested with nitric acid and hydrogen peroxide using commercial microwave system. Stannous chloride (1 % in 2.5 % HCl) was used as a reduction solution in CVG step. Method detection limit (MDL) was calculated according to the US-EPA 200.7 method and was found to be 0.0235 μg g⁻¹ for Hg I 253.652 nm. The effect of HCl concentration both in sample and in reduction solution, in addition with ICP nebulizer gas flow rate were tested to obtain high sensitivity. The highest sensitivity was obtained using a nebulizer gas flow rate of 0.5 L min⁻¹ and acid mixture of 5 mL HNO₃ and 2 mL HCl in a sample volume of 50 mL. Recovery of the mercury (94 – 99.7 %) was excellent for all the plastics analyzed.

<table>
<thead>
<tr>
<th>Certified plastic sample</th>
<th>Certified value (mg kg⁻¹)</th>
<th>Measured value (mg kg⁻¹)</th>
<th>Recovery %</th>
</tr>
</thead>
<tbody>
<tr>
<td>PE – ERM-EC681k</td>
<td>23.7 ± 0.8</td>
<td>22.8 ± 1.5</td>
<td>96.2</td>
</tr>
<tr>
<td>PP – NMIIJ 8133-a</td>
<td>949.2 ± 19.6</td>
<td>946 ± 15</td>
<td>99.7</td>
</tr>
<tr>
<td>PVC – NMIIJ 8123-a</td>
<td>937.0 ± 19.4</td>
<td>890 ± 30</td>
<td>95.0</td>
</tr>
<tr>
<td>ABS – BAM H010</td>
<td>415.0*</td>
<td>390 ± 30</td>
<td>94.0</td>
</tr>
</tbody>
</table>

*Informative value, n = 6 for microwave digestion
ADVANTAGES OF A HIGH TEMPERATURE TORCH INTEGRATED SAMPLE INTRODUCTION SYSTEM OVER A DESOLVATION SYSTEM FOR THE ANALYSIS OF MICROSAMPLES THROUGH ICP-MS

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Inductively coupled plasma mass spectrometry (ICP-MS) has become a widely used analytical tool. However, matrix components can lead to spectral and/or non-spectral interferences. Moreover, matrix can affect the aerosol characteristics; thus modifying the total mass of analyte transported towards the plasma and the plasma performance. In the particular case of liquid microsamples, the use of low consumption highly efficient sample introduction systems is required. Alternatively, a desolvation system can be employed to lower the solvent mass delivered to the plasma thus mitigating interferences.

Three different sample introduction systems were compared in terms of sensitivity and detection limits for the analysis of liquid microsamples by Inductively Coupled Plasma Mass Spectrometry (ICP-MS). The sample introduction systems evaluated were a high efficiency pneumatic concentric micronebulizer attached to either a Scott spray chamber, a desolvation system (Apex-HF) or a single pass spray chamber (hTISIS). The sample introduction was carried out in continuous as well as in segmented injection modes.

The results revealed that in both sample aspiration modes, higher sensitivities were obtained with the hTISIS and Apex as compared to a conventional liquid sample introduction system. Besides, the highest sensitivity was obtained when the temperature of the single pass spray chamber was set at values above 150°C. Moreover, lower limits of detection were obtained with hTISIS and Apex devices. Regarding memory effects in continuous mode, the wash-out times were 6 s, 40 s and 55 s for hTISIS at high temperature, the reference system and the Apex, respectively.

Matrix effects were also studied with different samples. For this purpose, spiked real samples were analyzed. The results revealed that the recoveries were closer to 100% for the hTISIS at high temperature than for the Apex and the double pass spray chamber.
(P-20)
LASER ABLATION COUPLED TO ICP TECHNIQUES FOR THE ANALYSIS OF CATALYSTS

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Heterogeneous catalysts contain metals deposited on large alumina or alumino-silicates surfaces. Their properties and status are correlated with the concentration of these metals. A good approach to perform elemental determination in this kind of samples is to adapt a laser ablation (LA) system to an Inductively Coupled Plasma Spectrometer (ICP-OES or ICP-MS). However, one of the most important drawbacks of LA is related with the calibration step. In this case, external calibration using matrix-matched solid standards is normally recommended. Unfortunately, matrix-matched solid standards are often not available. Therefore, a new calibration method is necessary.

In the present work, several alumina based catalysts were analyzed using LA-ICP techniques. The laser ablation system was a Q-switched Nd:YAG laser operated at 213 nm. In order to minimize fractionation which leads to a non-stoichiometric subsampling, an optimization of laser parameters was carried out for all the elements. Besides, results corresponding to crater and aerosol characterization by means of Secondary Electron Microscopy are discussed.

Regarding the calibration step, a dried droplet calibration method is suggested for carrying out quantitative analyses without using matrix-matched solid standards. This method is based on the deposition of several droplets, containing increasing elemental concentrations, on the sample surface. Once the solvent of the droplets is evaporated, the dried solid deposits are ablated. This technique could be considered as a simple standard addition methodology for laser ablation.
(P-21)
DETERMINATION OF RUBIDIUM IN DIFFICULT MATRICIES BY INDUCTIVE COUPLED PLASMA OPTICAL EMISSION SPECTROMETRY

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When analyzing low concentrations of rubidium in difficult matrix by inductive coupled plasma optical emission spectrometry (ICP-OES), analysis can suffer a lot of interference. Although axial plasma viewing is more prone to interferences than radial, axial measurement is the only option to reach the required sensitivity. Multilinear regression (MLR) method was used to correct for the interferences observed in axial measurement of rubidium, caused by other alkali and earth alkali metals.

For MLR method 19 synthetic samples were prepared at five levels of concentrations: rubidium 1-5 mg L⁻¹ and interfering elements Na 15-1615 mg L⁻¹, K 0.5-50.5 mg L⁻¹, Ca 10-1010 mg L⁻¹, Mg 5-55 mg L⁻¹, Fe 0.2-2.2 mg L⁻¹. Concentrations of rubidium and interfering elements were measured and results were compared to the added values. Measured concentrations of rubidium were 22-189% higher in all synthetic samples compared to added rubidium concentrations. From the suspected interference elements only sodium and calcium had significant effect on the determined concentrations of rubidium. After correcting measured rubidium values with MLR model, according to t-test measured and added rubidium concentrations had no significant difference at 95% level of confidence. Correction model worked best with high concentrations of sodium, but with low sodium concentration the correction was too severe and the corrected values were too low. Determination of low rubidium concentrations was also troublesome; after the correction rubidium concentration were still 20-57% higher than added values. The effect of calcium on measured rubidium concentration was minor compared to sodium.

MDL method worked quite well with synthetic samples when rubidium concentrations were 2-5 mg L⁻¹. With low rubidium concentrations (1 mg L⁻¹) more optimization is still needed.
DEVELOPMENT OF AN LC-ICP-MS METHOD FOR ZINC SPECIATION IN FISH FEEDS

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In analytical chemistry, speciation is considering individually the different chemical species of an element. Information regarding speciation is very important since the biological role of any particular element greatly depends on its chemical form. Over the past few decades, novel protein sources have been evaluated with the purpose to replace fish protein and fish oil in fish feeds. Nowadays, fish feeds are largely plant-based, which implies the need to adjust the content of amino acids, minerals and vitamins to meet the requirement of the fish. Zinc is an important micromineral in biology and it plays an essential role in fish nutrition [1]. Fish meal is the major source of zinc in fish feeds and following the increased use of plant materials there is a need to supplement with zinc to meet the requirement. Different inorganic and organic zinc forms are approved by the European Commission to be used as feed additives [2]. However, the knowledge on the availability of the different zinc compounds is lacking and there is a need to get more data on zinc bioavailability and to understand the role of different zinc species in fish nutrition. The project “Apparent availability and requirements of microminerals in salmon”, which is funded by the Norwegian Research Council (grant no. 244490) will provide some data in order to overcome this gap. Speciation methods using LC-ICP-MS will be developed to identify and quantify zinc compounds present in feed ingredients, feed and fish tissues from a feeding trial with Atlantic salmon. Different types of chromatography will be tested, such as ion chromatography, reversed phase and size-exclusion chromatography in order to separate zinc species. Considering the list of approved zinc-containing feed additives, standards of inorganic forms of zinc (e.g., zinc chloride and zinc oxide) and organic-bound compounds of zinc (e.g., zinc chelates of methionine or hydroxy analogues of methionine) will be used for the method development and optimization. A multivariate experimental design analysis will be applied for an efficient development and optimization of the analytical method. The poster will present an outline of the experiments planned and preliminary results.

References:

Acknowledgments:
This work is part of the project “Apparent availability and requirements of microminerals in salmon”, which is funded by the Norwegian Research Council (grant no. 244490).
DETERMINATION OF PHOSPHORUS, ALUMINUM AND IRON FROM SYNTHETIC MUNICIPAL WASTE WATER SLUDGE LEACHATE SAMPLES BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETRY ANALYSIS

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Abstract

The concentrations of phosphorus, iron and aluminum were determined from an acidic leachate of synthetic municipal waste water sludge. Hydrochloric acid was used to leach synthetic municipal waste water sludge which contained phosphorus, aluminum and iron.

The measurements were performed with PerkinElmer Optima 8300 ICP-OES – spectrometer. The GemCone low flow – nebulizer with cyclonic spray chamber was used for sample introduction. The determination of element concentrations was performed with a plasma gas flow of 8 L min\(^{-1}\), auxiliary gas flow of 0.2 L min\(^{-1}\), nebulizer gas flow of 0.6 L min\(^{-1}\) and plasma power of 1500 W. Axial measurement was used for iron and aluminum and radial measurement for phosphorus. The element concentrations were determined at wavelength 396.153 and 238.204 for aluminum and iron, respectively and for phosphorus at wavelength 213.617.

The results obtained were accurate and precise, so it can be said that ICP-OES is a good method for analyzing the concentration of iron and aluminum from an acidic leachate of synthetic municipal waste water sludge. Measurement parameters and calibration data will be presented more detailed in the poster.
ICP-OES FOR QUALITATIVE AND QUANTITATIVE ANALYSIS OF MINERAL ELEMENTS (P, K) IN THEIR RECOVERY FROM FLY ASH

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Correlation between the concentrations of elements having fertilizer properties such as P and K with particle size in the fly ash from Finnish power plants were examined with a viewpoint to obtain fractions suitable for forest fertilization. Fly ash samples (n=7) were sieved into five fractions. Wet chemical leaching (with HCl & HF) followed by ultrasound-assisted digestion was used as sample pre-treatment method. Thus prepared samples were analyzed by Perkin Elmer Optima 8300 ICP-OES instrument equipped with a Scott type spray chamber and a cross flow nebulizer. In addition to P and K, concentration of matrix elements like Al, Ca, Fe, Mg, Mn, Na, Si & Ti and heavy metal such as As, Cd, Cr, Cu, Ni, Pb & Zn were determined.

Figure 1. Relative distribution of (a) phosphorus (b) potassium in particle size fractions (♦ represents individual data point for n=7, box plot represents variation of P & K composition within the fractions)

Fractionation of fly ash attributes to increase in P and K concentration in smaller (<45-63 µm) size fractions by 1.3-2 and 1.3-1.6 percentage unit, respectively. Fractionation of fly ash is a potential method to improve the fly ash chemistry that is suitable for forest fertilization. However, the concentration of heavy metal in the fly ash fraction remains a criterion for the acceptance of such fraction as forest fertilizer products.

There is a Need for Speciation Analysis of Selenium in Fish Feed and Fish Tissue

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Selenium (Se) is an essential, but also potentially toxic, trace element. Seafood and marine raw materials have natural high levels of Se and are important sources of Se for both humans and farmed animals. For fish, Se is important e.g. for the antioxidant defense and thyroid metabolism. Selenium can be present in different chemical forms, or Se species, categorized as inorganic and organic Se species. The bioavailability and toxicity of Se is dependent on the physiochemical properties of the Se species present.

Fish feed contains relative high levels of Se, typically ranging from 0.2 to 9 mg/kg feed [1,2]. In the current EU feed regulation the maximum level for total Se in fish feed is set at 0.5 mg/kg feed (Council Directive 70/524/EC and amendments). The levels of Se in fish feed depend on the levels of Se in the feed ingredients, e.g. natural high levels of Se in the fish meal causes high levels of Se in the complete feed. In the aquaculture industry, however, there has been a shift towards replacement of marine-based ingredients, such as fish meal, with plant based ingredients in the feed. This practice changes both the Se levels and the Se speciation profile. Since the bioavailability of Se is highly dependent on the species present, there is a need to perform Se speciation analysis of fish feed, as well as of the feed ingredients, to determine the major Se species present. Furthermore, Se speciation analysis of fish that have been fed different feeds, such as plant-based feed, marine-based feed and supplemented feed (addition of e.g. Se-containing yeast) will provide important information on how the feeds may affect the Se levels and species in the fish. Selenium speciation analysis of fish feed and of fish tissue is hence an important tool in the assessment of bioavailability of Se in fish feeds.

Selenium speciation of fish feeds and muscle tissue of Atlantic salmon were conducted using High Pressure Liquid Chromatography coupled to Inductively Coupled Plasma Mass Spectrometry (HPLC-ICPMS). The sample extraction procedure and the chromatographic separation methods were optimized during the method development. The methods have been validated, and have been applied to feed and salmon tissue. Preliminary results from a feeding trial with Atlantic salmon being fed different levels and sources of Se in the fish feed will be presented.


ELUCIDATION OF THE NEGATIVE MODE IONIZATION MECHANISM OF PERFLUORINATED COMPOUNDS USING AN ACTIVE CAPILLARY PLASMA IONIZATION SOURCE

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Introduction
Active capillary plasma ionization is a soft ionization method developed in our laboratory, which is based on a dielectric barrier discharge. The cold plasma source itself serves as an interface to the mass spectrometer. It features a fully enclosed ionization volume and precisely controllable gas-phase conditions, which facilitates mechanistic investigations. In this study, perfluorinated compounds (PFC) were investigated, since these compounds are challenging to analyse with other ambient ionization techniques. It is interesting to understand why our source is capable of ionizing these compounds. The elucidation of the negative mode ionization mechanism of perfluorinated compounds would allow us to better understand the gas-phase chemistry of our source, compare it with other ambient ionization techniques and determine useful applications relying on the specific features of our source.

Methods
A range of perfluorinated compounds (including perfluoroheptane, perfluorotributylamine) were investigated. A pressure-assisted nanospray setup was used to generate a defined gas-phase analyte concentration. The gas-phase sample generation system consisted of a pressurized sample reservoir, which is connected via a fused silica capillary to a hollow heating cartridge held at 200 °C. The sample flow delivered via the capillary was evaporated within the cartridge and a preheated plasma gas stream (air or N₂, 2 L/min) was used to transport the sample to the source. The gaseous sample was ionized by an active capillary plasma ionization source that was directly connected to the inlet of the mass spectrometer. The active capillary plasma ionization source contained a stainless steel capillary inserted into the glass capillary serving as one electrode and a copper ring surrounding the glass capillary functioning as the counter-electrode. The cold plasma was generated by applying a sine wave modulated (5750 Hz) high voltage (1.0 – 3.0 kV, peak to peak) to the electrodes. For all measurements, a commercial ion trap instrument (LTQ Finnigan, Thermo Fischer) was used for the detection.

Results and Discussion
The main gas-phase reaction products of perfluorinated alkanes were a loss of one fluorine atom [M-F]- and a substitution reaction of fluorine by oxygen [M-F+O]-. The radical anion [M]- was only detected by applying lower voltage (1.0 kVpp). It was found that the applied voltage has a great impact on negative ion formation. The oxygen source for the substitution reaction was successfully determined by operation under different conditions. NO₂ and NO₃ ions were generated in the plasma source, but it is not clear if they react with the perfluorinated alkanes. H₂O and O₂ molecules present (in traces) in the plasma-gas were identified to contribute as oxygen sources for the substitution reaction.

Conclusions
Our results clearly show that the active capillary plasma ionization source is capable of ionizing perfluorinated compounds. The ionization efficiency can be further increased through the knowledge gained about the ionization mechanism of perfluorinated compounds. We are presently determining the nature of all the reactive species, which will allow us to further improve the sensitivity and specificity as well as discover novel applications for the source.
(P-27)
DEVELOPMENT OF A CHIP-BASED GRAPHENE-TITANIUM DIOXIDE NANOCOMPOSITE-ASSISTED PHOTOCATALYTIC REDUCTION DEVICE TO COUPLE HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY WITH INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY FOR ONLINE DETERMINATION OF INORGANIC SELENIUM SPECIES

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In this study, we developed a chip-based vapor generation (VG) system to interface chromatographic separation with inductively coupled plasma-mass spectrometry for the determination of inorganic selenium (Se) species. The chip-based VG system consisted of a burgeoning photocatalyst graphene-titania dioxide (GR-TiO₂) nanocomposite and a poly(methyl methacrylate) (PMMA)-based photocatalyst-assisted reduction device.¹ Because the used nanocomposite possessed extended light absorption ability and greater charge separation efficiency,² efficient conversion of Se(IV) and Se(VI) into volatile species was expected. Additionally, due to the concept of lab-on-a-chip employed in the system design, operational functionalities such as sample/reagent mixing and photocatalyst-assisted reduction were integrated onto a minute substrate, leading to simple operation.³ Based on the analytical results, the proposed hyphenated system was proven to be a promising platform for online determination of inorganic selenium (Se) species.

Reference:
ANALYSIS OF GAS TEMPERATURE AND OXYGEN EMISSION IN HIGH FREQUENCY ELECTRODELESS LIGHT SOURCES

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Preparation of the light sources for different scientific devices is always connected with studies of plasma conditions inside the lamp. The top-choice diagnostic methods are those that give information without disturbing the plasma – so called non-invasive methods. One of such methods is determination of gas temperature from rotational spectra of molecules. Depending on the filling and conditions inside the light source, different molecules can be formed in the plasma. If they are formed in places with different conditions (for instance, difference in gas temperatures) they will indicate diverse values of rotational temperature. For instance, it has been observed that C₂ shows higher values of rotational temperature than OH radical, this difference could be connected with the fact that C₂ is formed in the central part of the plasma, but OH is formed near the walls where hydrogen atoms combine with oxygen coming from the walls.

In this work we present results of gas temperature determination from OH radical rotational spectra for helium- and hydrogen-containing light sources and analysis of oxygen atomic line emission. The light sources under study were high-frequency electrodeless lamps (HFEDLs) filled with helium and hydrogen mixture (pressure ~1 Torr). These HFEDLs are of cylindrical form with diameter of 2 cm and length 4 cm. The discharge is ignited by placing lamp into the electromagnetic field of 100 MHz frequency. The emission from lamp was registered using JobinYvon SPEX 1000M spectrometer for different excitation generator current values (100 mA – 200 mA).

Taking into account that light sources are filled with hydrogen and helium, they should not contain any other elements; however in emission spectra we could register atomic lines of oxygen. When increasing the value of current, the intensity of oxygen lines increased as well as OH emission intensity and rotational temperature (determined from the OH spectra).

Experimental results support the hypothesis that OH radical is formed near the walls of the lamp and it is also excited there, too.

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(P-29) 
ESTIMATION OF ELECTRON TEMPERATURE FROM EMISSION OF ARGON IN MULTI-ELEMENT CADMIUM AND ZINC LIGHT SOURCES

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Multi-element light sources can be of interest for atomic absorption spectroscopy due to possibility to measure concentrations of pollutants in the sample almost simultaneously without changing light source. Before putting the lamp into analytical device it must undergo the optimization process to find the best operating regime and most appropriate filling. To reach the goal, information about the plasma conditions inside the lamp is very helpful. In this study we present results of electron temperature determination in multi-element light sources containing zinc and cadmium.

The light sources under study were high-frequency electrodeless lamps (HFEDLs) filled with cadmium and zinc (with argon as buffer gas). The lamps were chosen with slightly different fillings: (1) Zn+Cd+Ar (p = 3 Torr) and (2) Zn+Cd+SbI3+Ar (p = 2.7 Torr). These lamps are prototypes for use in atomic absorption spectrometers, particularly, Lumex MGA-915M. These HFEDLs consist of spherical part (with diameter of 1cm) and small side-arm. The discharge is ignited at the spherical part of the lamp by placing it into the electromagnetic field of 100 MHz frequency. The emission from lamps was registered using JobinYvon SPEX 1000M spectrometer for different excitation generator voltages (21V – 27V).

Electron temperature was determined using emission spectra of argon atom. In this study it was assumed that electron energy distribution follows Maxwell-Boltzmann distribution, and excited levels are mostly populated by collisions between electrons and atoms in the ground state. For chosen atomic lines of argon the electron impact excitation rate coefficients were used in exponent approximation. Spectroscopic data together with measured intensities of spectral lines was used to prepare Boltzmann plots. Electron temperature was then determined from the slope of these plots.

The analysis of the experimental results showed that the estimated electron temperature in both types of the lamps was around 0.5 eV and remained almost constant in the excitation generator voltage range from 21V to 27V. For lamps containing Zn and Cd value of electron temperature was around 0.45 eV, and for lamps with added SbI3 it was slightly higher – above 0.5 eV.
THE POTENTIAL OF DUAL PROBE ANALYSIS

Dick Waijers

Shell Technology Centre Amsterdam, Netherlands

Sample introduction for ICP applications is mainly done by making use of a peristaltic pump. Addition of an internal standard using a Y-piece and an additional peristaltic pump channel is rather common. Other uses like the on-line addition of an isotopically-enriched solution have also been reported. Yet there are more possibilities using a second channel of the peristaltic pump, hereafter referred to as dual probe analysis. The applications discussed are:

On line standard addition using the first probe for the sample and a second probe for the additions. This set-up avoids the need to make a lot of separate samples with additions whilst keeping the advantages of standard addition. Optional external calibration can still be performed when using a blank as sample.
Isotope ratio analysis with in-situ mass bias correction by adding one or more isotopic standards via the second probe.
Adding isotope dilution standards to blanks and samples, with in-situ mass bias correction and the possibility to add different concentrations of isotope dilution standard in one batch.
MULTIELEMENTAL ANALYSIS OF PURIFIED AND TAB WATER SAMPLES WITH HR ICP-MS

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Eight different water samples were analysed with High Resolution sector Field Inductively Coupled Plasma-Mass Spectrometer (HR ICP-MS, Element 2, ThermoScientific) in order to improve the reliability of HR ICP-MS analysis of very low levels of elements in water samples. Two parallel samples were collected and measured from each of the following waters: milliQ Integral 5 (Millipore), milliQ Advantage A10 (Millipore), Elix® Advantage 5 milliQ (Millipore), Research reactor (FIR 1) cooling water, cold (8 °C) and hot (52 °C) Finnish tab water, hot (77 °C) water from the automated coffee maker and certified Reference Material for Measurement of Elements in Surface Waters (SPS-SW2).

Elemental analyses were performed from undiluted and 1/10 diluted sample solutions. Calibration curve and control samples were diluted from ICP-MS Multi-Element Solutions 2 and 4 by SPEX and control samples were diluted from Semi-Quantitative Standard (SQS-1) AccuTrace™ and ICP Laboratory Performance Check Standard 1 (LPC-1) Reference standard solutions. 10 µg/L of indium was used as an internal standard in all samples, background, calibration and control samples. The samples were injected through SeaSpray nebulizer (0.4 ml/min) and double pass spray chamber equipped with Peltier cooling unit. Aluminium sample cone and Nickel skimmer cone were used during the measurement. Table 1 shows the measured elements, their limits of detection and the resolution of measurement.

Table 1. Detection limits (µg/L) for analysed elements in 1 % HNO₃. Low resolution (R<300) in green, medium resolution (R<4000) in orange and high resolution (R<10,000) in purple.

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<th>Ag-107</th>
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<td>0.013</td>
<td>0.03</td>
<td>0.02</td>
<td>0.03</td>
<td>0.005</td>
<td>0.6</td>
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| Measured concentrations of reference material samples were in a good accordance with the certified concentrations of the reference material. The difference between the measured concentrations and certified concentrations were less or equal to 10 % to almost all elements, except 26 % for Se. Concentrations in FIR 1 reactor cooling water were close to the concentrations in milliQ water samples. However, concentrations of B, Pb, P, Fe, Na and Si were significantly higher in FIR 1 water sample. Water temperature did not have significant effect on the concentrations of most elements in tab water samples. However Cd, Pb, Ni and Zn had higher concentrations in hot water than in cold water. Nevertheless, all elemental concentrations in tab water samples were below the Finnish tab water quality requirements.
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