11th Nordic Conference on Plasma Spectrochemistry

June 9 - 13, 2024
Loen, Norway

Programme and Abstracts

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2 Welcome Letter

Dear colleagues,
On behalf of the Analytical Section of the Norwegian Chemical Society and the Organising Committee it is a great honour and pleasure to welcome you to Loen and the 11th Nordic Conference on Plasma Spectrochemistry. This conference aims to provide both an international and regional forum by which researchers and users can share their knowledge and exchange ideas about recent developments and state-of-the-art knowledge in main areas of plasma spectrochemistry. The conference brings together participants experienced in fundamental aspects, instrumentation and applications to examine recent progress in this continuing growing field; 120 participants are participating in this 2024 conference.

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 are present, the conference will hopefully also be a stimulating forum for communication across borders and between scientific disciplines.

In contrast to other plasma spectrochemistry meetings, short tutorial courses are offered by leading scientists to all participants during the four and a half day meeting which may be attractive to all participants. This concept is meant to stimulate especially students and Nordic users of plasma-based techniques to learn more about theoretical and practical issues as well as to obtain specialists recommendations for method improvements and novel applications.

There’s something extraordinary about meeting *in-person*. Great minds coming together in one place fuels creative energy and innovation, but we also know that the natural beauty of the area will captivate you.
We sincerely 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!

On behalf of the Organising Committee

Yngvar Thomassen
Conference Chair

3 Organising and Scientific Committee

Yngvar Thomassen	(Conference Chair), National Institute of Occupational Health, Oslo and Norwegian University of Life Sciences, Ås, Norway
Arne Bengtson	Swerim, Kista, Sweden
Balazs Berlinger	University of Veterinary Medicine, Budapest, Hungary
Elin Gjengedal	Norwegian University of Life Sciences, Ås, Norway
Walter Lund	University of Oslo, Norway
Ivar Martinsen	(Treasurer) Norwegian Chemical Society, Oslo, Norway
Oddvar Røyset	Norwegian Chemical Society, Oslo, Norway
Jens Sloth	Technical University of Denmark, National Food Institute, Denmark
Ari Väisänen	University of Jyväskylä, Finland
Arne Åsheim	(Exhibition Coordinator), Norwegian Chemical Society, Porsgrunn, Norway

4 General Information

Conference Desk

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

Saturday	June 8	15:00 - 18:00
Sunday	June 9	08:00 - 09:00
Monday	June 10	07:30 - 08:00
Tuesday	June 11	07:30 - 08:00
Wednesday	June 12	07:30 - 08:00
Thursday	June 13	08:00 - 09:00

Participants are requested to register as soon as possible upon arrival to the conference venue, 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 8, 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 SpectraPure Standards AS.

Sunday, June 9, 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. You will be back at Hotel Alexandra around 19:30.

Sunday, June 9, 14:15: A visit to Kjenndalen and Kjenndalsbreen

Kjenndalen and Kjenndalsbreen glacier start at the head of the Loen Valley. By bus you will travel up Loen Valley to Kjenndalen almost all the way to the glacier. The Kjenndalsbreen glacier is located innermost at the bottom of the valley, 17 km from Loen. It's a 15-minute walk from the car park to the lowest glacier arm of the Jostedalsbreen. The glaciers grew more than 300 metres between 1980 to 1997 but have receded in recent years. You will be back at Hotel Alexandra around 17:30.

Sunday, June 9, 14:15: Mountain hiking for fit spectroscopists: The Skåla Challenge

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

Sunday, June 9, 14:15: Loen Skylift

Loen Skylift is a spectacular attraction and adventure arena in the inner part of the Nordfjord. A cable car will lift you from the fjord to 1011 m above sea level. Here you can enjoy the views of the fjord landscape – from the restaurant table, or while exploring in the mountains.

Monday, June 10, 21:00: Bring your own wine or beverage to the poster viewing and wine and beverage tasting

The cork fee charged by the hotel to allow you to bring your own alcohol containing beverage is kindly *sponsored by Holger Hartmann*.

Wednesday, June 12, 16:40: Excursion to the Briksdal Glacier with a conference outdoor dinner

You are invited to visit the heart of Norway - an **Unforgettable** trip to the Briksdal Glacier. Participants can enjoy a walk to view the enormous cascade of ice where you will be able to test the best aquavit in the world; *Gilde Aqua Ultra Plus*, or the local *Loen Apple Juice*
kindly sponsored by 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 once again be in charge of the kitchen to ensure your best outdoor food experience.
This event is kindly sponsored by NU Instruments Ametek and Massanalys Spectrometry Nordic.

6 Scientific Programme

Oral presentations
Invited plenary lectures and submitted oral contributions will be 30 and 20 minutes in length, respectively (including discussion).
Video projectors will be provided in all lecture rooms.

Posters
The posters should be mounted early Sunday morning June 9, 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.

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:

SPS Spectrapure Standards
JAAS
NKS
HOLGER HARTMANN
nu / AMETEK®
matriks KJEMISK TEKNOLOGI
msnordic

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

The following companies have registered for display and demonstration:

Holger Hartmann AS / Milestone/Spectro
Houm AS/Anton Paar
Labkings B.V.
Matriks AS/Agilent Technologies/Inorganic Ventures
Narema
Nu Instruments Ametek
PerkinElmer
Shimadzu Europa GmbH
Thermo Fisher Scientific

9 Correspondence after the conference

Yngvar Thomassen
National Institute of Occupational Health
Gydas vei 8, 0369 Oslo, Norway
Tel: +47 23 10 53 20 +47 99 51 05 21
E-mail: Yngvar.Thomassen@stami.no


Anton Paar
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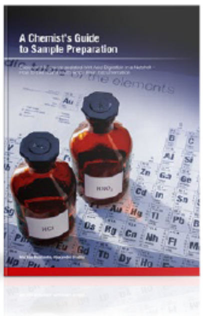
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The flagship SpectroARCOS

We also offer standards made in Norway from Spectrapure Standards: Single element, Multi elements and Custom Made.

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10 Schedule of events

Saturday, June 8, 2024

15:00 - 18:00	**Registration**
17:00 - 19:00	**Informal get-together in the Hotel Alexandra Bath & Spa** *This event is kindly sponsored by SpectraPure Standards AS*
From 19:00	**Dinner**

Sunday, June 9, 2024

08:00 - 09:00	**Registration**
09:00 - 09:15	**Welcome and opening remarks**
09:15 - 11:15	**Session I: Progress in plasma spectrochemistry**
11:15 - 11:45	**Coffee break, poster viewing and exhibition**
11:45 - 12:45	**Session I continues: Progress in plasma spectrochemistry**
13:00 - 14:00	**Lunch**
14:00 -	**Excursion to Geiranger, The Skåla Challenge, Loen Skylift and Kjenndalen**
From 19:00	**Dinner**

Monday, June 10, 2024

08:00 - 10:20	**Session II: Plasma spectrochemistry in speciation analysis**				
10:20 - 10:50	**Coffee break, poster viewing and exhibition**				
10:50 – 12:50	**Session II continues: Plasma spectrochemistry in speciation analysis**				
13:00 - 14:00	**Lunch**				
14:00 - 15:30	**Session III: Plasma spectrochemistry and imaging**				
	Short Course Session I				
15:45 - 17:30	**Short course A1**	**Short course A2**	**Short course A3**	**Short course A4**	**Short course A5**
17:30 – 18:00	**Coffee break, exhibition and poster viewing**				
	Short Course Session II				
18:00 -19:45	**Short course B1**	**Short course B2**	**Short course B3**	**Short course B4**	**Short course B5**
From 19:00	**Dinner**				
21:00	**Poster viewing and discussions with wine and beverage tasting** **(Bring your own wine or beverage)** *This event is kindly sponsored by HolgerHartmann.*				

Tuesday, June 11, 2024

08:00 - 09:50	**Session IV: Advances in isotopic applications and measurements**
09:50 - 10:20	**Coffee break, poster viewing and exhibition**
10:20 - 12:30	**Session IV continues: Advances in isotopic applications and measurements**
12:30 - 13:30	**Lunch**
13:30 - 15:30	**Session V: Vendor contributions**
	Short Course Session III
15:45 - 17:30	**Short course C1** **Short course C2** **Short course C3** **Short course C4** **Shor course C5**
17:30 - 18:00	**Coffee break, poster viewing and exhibition**
	Short Course Session IV
18:00 - 19:45	**Short course D1** **Short course D2** **Short course D3** **Short course D4**
From 19:00	**Dinner**

Wednesday, June 12, 2024

08:00 - 10:10	**Session VI: Laser ablation applications**
10:10 - 10:40	**Coffee break, poster viewing and exhibition**
10:40 - 11:40	**Session VI continues: Laser ablation applications**
11:50 - 13:00	**Session VII: Bio-imaging and single cell/particle analysis**
13:00 - 14:00	**Lunch**
14:00 - 16:20	**Session VII continues: Bio-imaging and single cell/particle analysis**
16:40	**Departure for the excursion to the Briksdal Glacier with a conference outdoor dinner**

Thursday, June 13, 2024

08:30 - 09:40	**Session VIII: Applications of plasma spectrochemistry in food, material and environmental sciences**
09:40 - 10:10	**Coffee break**
10:10 - 11:30	**Session VIII continues: Applications of plasma spectrochemistry in food, material and environmental sciences**
11:30	**Closing remarks and farewell**
12:00	**Lunch**

11 Daily Programme

Sunday, June 9, 2024

Time	Abstr.	
09:00- 09:15		**Welcome/Opening remarks** Yngvar Thomassen, Conference chair
		Session I: Progress in plasma spectrochemistry Chair: Joanna Szpunar
09:15- 09:45	**O-1**	**Distinguished Speaker Lecture: Advanced techniques in plasma spectrochemistry: Origins and future** Gary M. Hieftje *Indiana University, Bloomington, IN, USA*
09:45- 10:15	**O-2**	**Acoustic fields to shape, move, and gate ion beams for mass spectrometry** Jacob T. Shelley,[1] Yi You,[2] Julia L. Danischewski,[1] Lauren S. Bauer,[1] and Jens Riedel[2] [1]*Department of Chemistry and Chemical Biology, Rensselaer Polytechnic Institute, Troy, NY, USA* [2]*Department of Instrumental Analytics, Federal Institute for Materials Research and Testing (BAM), Berlin, Germany*
10:15- 10:45	**O-3**	**Advancements in elemental and molecular mass spectrometry for the characterization of nanoparticles and macroplastics** Carsten Engelhard[1,2], Annika Schardt[2], Johannes Schmitt[2], Cristian C. Escobar-Carranza[2], Désirée A.-M. Schütz[2], Manuel Heinelt[2] [1]*BAM, Bundesanstalt für Materialforschung und -prüfung, Berlin, Germany* [2]*University of Siegen, Department of Chemistry and Biology, Siegen, Germany*
10:45- 11:15	**O-4**	**Quantitative nano depth profiling by means of high-energy laser desorption ionization (HELDI)** Davide Bleiner[1,2], Sharath Rameshbabu[1,2] and Janosch von Ballmos[2] [1]*Department of Chemistry, University of Zurich, Switzerland* [2]*Empa – Swiss Federal Laboratories for Materials & Technology*
11:15- 11:45		**Coffee, exhibition and poster viewing** Chair: Jacob T Shelley
11:45- 12:05	**O-5**	**25 years of the dynamic reaction cell - Predictable reactions across the entire periodic table** Minyu Zuo *(PerkinElmer)*, Michael Petrich *(PerkinElmer) and* Karl Andreas Jensen *(Norwegian University of Life Sciences NMBU, Centre for Environmental Radioactivity CERAD)*
12.05- 12:25	**O-6**	**Coupling laser ablation SF-ICP-MS, Q-ICP-MS, MC-ICP-MS and ICP-OES with electron microscopy for interdisciplinary (geology-biology-archaeology) research** Tonny B. Thomsen, Benjamin D. Heredia, Sebastian N. Malkki and Nynke Keulen *The Geological Survey of Denmark and Greenland (GEUS), Copenhagen, Denmark*

Time	Abstr.	
12:25- 12:45	**O-7**	**Advancements in elemental analysis of lithium ion batteries using ETV-ICP-OES – Challenges and opportunities** Dennis Kessen[1], Matthias Walter[1], Martin Winter[1,2], Sascha Nowak[1], Simon Wiemers-Meyer[1], *[1]University of Münster, MEET Battery Research Center, Germany* *[2]Helmholtz-Institute Münster, IEK-12, FZ Jülich, Münster, Germany*
13:00		**Lunch**
14:00		**Excursion to Geiranger, the Skåla Challenge, Kjenndalen, and Loen Skylift**
From 19:00		**Dinner**

Monday, June 10, 2024

Session II: Plasma spectrometry in speciation analysis

Chair: Jörg Feldmann

Time	Abstr.	
08:00- 08:30	**O-8**	**Plasma mass spectrometry in speciation analysis - Present and future** Joanna Szpunar *IPREM, UMR5254 CNRS-UPPA, Pau, France*
08:30- 09:00	**O-9**	**A closer look to the elemental world of fungi with HPLC coupled to ICP-MS** Walter Gössler *University of Graz, Austria*
09:00- 09:20	**O-10**	**A new approach to speciation stabilization – Species separation at the sampling site** Beata Krasnodębska-Ostręga, Joanna Kowalska, Katarzyna Kińska and Monika Sadowska *Faculty of Chemistry, University of Warsaw, Poland*
09:20- 09:40	**O-11**	**Selenium speciation studies in cancer patients to evaluate the responses of biomarkers of selenium status to different selenium compounds** Christian Ward-Deitrich[1], M. Estela del Castillo Busto[1,2], Stephen O. Evans[3,4], Margaret P. Rayman[5], Michael B. Jameson[4,6] and Heidi Goenaga-Infante[1] *[1]LGC National Measurement Laboratory (NML), Queens Road, Teddington, Middlesex TW11 0LY, United Kingdom* *[2]Grupo Química Analítica Aplicada (QANAP), Instituto Universitario de Medio Ambiente (IUMA), Universidade da Coruña (UDC), A Coruña, Spain* *[3]Department of Biological Sciences, University of Waikato, Hamilton, New Zealand* *[4]Waikato Clinical Campus, University of Auckland, Hamilton, New Zealand* *[5]Department of Nutritional Sciences, University of Surrey, Guildford, UK* *[6]Oncology Department, Waitako Hospital, Hamilton, New Zealand*
09:40- 10:00	**O-12**	**Robust sample preparation for automated speciation analysis of gadolinium-based contrast agents in tissue samples** Torben J. Maas and Uwe Karst *Institute of Inorganic and Analytical Chemistry, University of Münster, Germany*

Time	Abstr.	
10:00 – 10:20	O-13	**Development of HILIC-ICP-MS method for pharmaceutical complexes of Pt, Gd and I monitoring in drinking water resources** Joseph Côme, Maïté Bueno, Florence Pannier and Sandra Mounicou *Universite de Pau et des Pays de l'Adour, CNRS, IPREM, Institut des Sciences Analytiques et de Physico-chimie pour l'Environnement et les Matériaux, Pau, France*
10:20-10:50		**Coffee break, exhibition and poster viewing** Chair: Jens J Sloth
10:50-11:20	O-14	**The importance of non-target screening and mass balances for PFAS analysis: a job for elemental analysis** Jörg Feldmann, Viktoria Müller, Eleonora Matic, Raquel Gonzalez de Vega, David Clases and Andrea Raab *Analytical Chemistry, University of Graz, Austria*
11:20-11:40	O-15	**Seeing beyond LC-MS/MS in fluorine analysis for ski waxes** Viktoria Müller [1,2], Raquel Gonzalez de Vega [2], Erwin Rosenberg [3], Klaus Zangger [4], David Clases [2], Andrew Kindness [1] and Jörg Feldmann [2] [1]*The James Hutton Institute, Craigiebuckler, Aberdeen, Scotland, UK* [2]*Analytical Chemistry, Institute of Chemistry, University of Graz, Austria* [3]*Institute of Chemical Technologies and Analytics, Vienna University of Technology, Austria* [4]*Organic Chemistry, Institute of Chemistry, University of Graz, Austria*
11:40-12:10	O-16	**From rare trace elements to plastics – Application of ICP-MS and complementary techniques to study environmental impacts of the Anthropocene** Daniel Pröfrock [1], Anna Ebeling[1,2], Lars Hildebrandt[1,2], Tristan Zimmermann[1], Dominik Wippermann[1,2], Alexa Zonderman[1,2], Ole Klein[1,2] [1] *Helmholtz-Zentrum Geesthacht, Abteilung Anorganische Umweltchemie, Geesthacht, Germany* [2]*Universität Hamburg, Department of Chemistry, Inorganic and Applied Chemistry, Germany*
12:10-12:30	O-17	**Evaluating the impact of soil composition on the mobility of tellerium species using ICP-MS** Katarzyna Kińska, Monika Sadowska and Beata Krasnodębska-Ostręga *Faculty of Chemistry, University of Warsaw, Poland*
12:30-12:50	O-18	**Mechanistic understanding and biomedical implications of d-electron metal - protein interactions in dairy proteins** Tetiana Dyrda-Terniuk[1], Oleksandra Pryshchepa[1], Kinga Robotnik[2,1] and Paweł Pomastowski[1,2] [1]*Centre for Modern Interdisciplinary Technologies Nicolaus Copernicus University in Torun, Poland* [2]*Department of Inorganic and Coordination Chemistry, Faculty of Chemistry, Nicolaus Copernicus University in Torun, Poland*
13:00 14:00		**Lunch**

Session III: Plasma spectrometry and imaging

Chair: Gary Hieftje

Time	Abstr.	
14:00- 14:30	**O-19**	**LIBS imaging: Recent advances and perspectives** Vincent Motto-Ros *Institut Lumière Matière, Université Lyon ,Villeurbanne, France*
14:30- 15:00	**O-20**	**Multimodal chemical imaging for biomedical applications** Uwe Karst, Katharina Kronenberg, Christine Verlemann, Peter Niehaus and Michael Sperling *Institute of Inorganic and Analytical Chemistry, University of Münster, Germany*
15:00- 15:30	**O-21**	**High-resolution bioimaging with nanoscale secondary ion mass spectrometry and complementary techniques** Dirk Schaumlöffel, Iris H. Valido and Maria Angels Subirana *CNRS/Université de Pau et des Pays de l'Adour, Pau, France*

Short Course Session I

15:45- 17:30	**A1-A5**	**A1** Vincent Motto-Ros	**A2** Johanna Irrgeher	**A3** Uwe Karst	**A4** Daniel Pröfrock	**A5** Jacob T. Shelley
		A brief introduction to LIBS	**Quality of measurements: Basics of method validation, traceability and uncertainty assessment**	**Multimodal imaging: Sample preparation, instrumentation and data evaluation**	**Hyphenated techniques for elemental speciation analysis in environmental samples – an overview**	**Electrical plasmas for molecular mass spectrometry**

17:30-
18:00 **Coffee break, exhibition and poster viewing**

Short Course Session II

18:00- 19:45	**B1-B5**	**B1** David Bleiner	**B2** Thomas J. Kozikowski	**B3** Dirk Schaumlöffel	**B4** Joanna Szpunar	**B5** Martin Šala
		Laser Induced XUV Spectroscopy (LIXS): The future of LIBS is now	**How to reduce ICP-OES interferences for 69 elements**	**SIMS and related techniques for elemental and isotopic imaging at micro- and nanometer scale**	**Complementarity of ICP-MS and electrospray MS/MS in advanced speciation analysis**	**LA-ICP-MS mapping; from fundamentals to the best image quality maps**

From
19:00 **Dinner**

21:00 **Poster viewing and discussions with wine and beverage tasting and exhibition.**
(Bring your own wine or beverage)

Tuesday, June 11, 2024

Session IV: Advances in isotopic applications and measurements

Chair: Uwe Karst

Time	**Abstr.**	
08:00-08:30	**O-22**	**Enhanced stable isotope tracking of elemental fluxes: from biological metabolism to industrial processes.** Johanna Irrgeher[1], Alexander Epov[1], Susanne Michelic[2,3], Markus Puschenreiter[4], Kathrin Thiele[2,3], Stefan Wagner[1], Andreas Zitek[5,6] and Thomas Prohaska[1] *[1]Johanna Irrgeher, Montanuniversität Leoben, Chair of General and Analytical Chemistry, Leoben, Austria* *[2]Montanuniversität Leoben, Chair of Ferrous Metallurgy, Leoben, Austria* *[3] Christian Doppler Laboratory for Inclusion Metallurgy in Advanced Steelmaking, Montanuniversität Leoben, Leoben, Austria* *[4]University of BOKU Vienna, Institute of Soil Research, Tulln, Austria* *[5]University of Natural Resources and Life Sciences Vienna, Department of Chemistry, Vienna, Austria* *[6] FFoQSI GmbH, Technopark 1D, 3430 Tulln an der Donau, Austria*
08:30-09:00	**O-23**	**Recent advances in isotopic analysis using MC-ICP-MS: Analytical method developments and applications** Lana Abou-Zeid[a], Laura Suárez-Criado[a,b], Maria Alessia Vecchio[a,c], Kasper Hobin[a], Marta Costas Rodríguez[d], Eduardo Bolea-Fernandez[a,e], Marco Grottic and Frank Vanhaecke[a] *[a] Ghent University, Department of Chemistry, Atomic & Mass Spectrometry – A&MS research group, Ghent, Belgium* *[b] University of Oviedo, Department of Physical and Analytical Chemistry, Julian Claveria 8, 33006-Oviedo, Spain* *[c] University of Genoa, Department of Chemistry and Industrial Chemistry, Italy* *[d] Centro de Investigación Mariña, Universidade de Vigo, Departamento de Química Analítica y Alimentaria, Grupo QA2, 36310 Vigo, Spain* *[e] University of Zaragoza, Aragon Institute of Engineering Research (I3A), Department of Analytical Chemistry, Zaragoza, Spain*
09:00-09:30	**O-24**	**Drug counterfeits – identification via element isotope ratios and trace elemental patterns** Stefan Stürup[a] and Else Holmfred[b] *[a]Department of Pharmacy, University of Copenhagen, Denmark* *[b]Green Earth Sciences, Stanford University, California, USA*
09:30-09:50	**O-25**	**Development and comparison of analytical methods for the simultaneous measurement of Sr and Pb isotope ratios** Federica Bruschi [a], A. Lores Padin [b], E. Bolea-Fernandez [c], K. Hobin [b], D. Cappelletti [a], F. Vanhaecke [b] *[a]Department of Chemistry, Biology and Biotechnology, University of Perugia, Italy* *[b]Department of Chemistry, Atomic & Mass Spectrometry - A&MS Research Group, Ghent University, Belgium* *[c]Department of Analytical Chemistry, Aragon Institute of Engineering Research, University of Zaragoza, Spain*

Time	Abstr.	
09:50- 10:20		**Coffee break, exhibition and poster viewing** Chair: Carsten Engelhard
10:20- 10:50	**O-26**	**From theory to practice: Implementing cutting-edge ICP-MS for swift radiochemical analysis.** Jixin Qiao *Department of Environmental and Resource Engineering, Technical University of Denmark (DTU Sustain), DTU Risø Campus, Roskilde, Denmark*
10:50- 11:10	**O-27**	**Tracing landfill leachate in environmental samples using a triple isotope approach - $\delta^{13}C$, $\delta^{11}B$ and $^{87/86}Sr$ in Norwegian landfill leachate** Christian Schöpke[1,2] Ingar Johansen[1], Vani Devegowda[1] and Julianne Hem[2] [1]*Institute for Energy Technology (IFE), 2007 Kjeller, Norway* [2]*Norwegian University of Life Sciences (NMBU), Ås, Norway*
11:10- 11:30	**O-28**	**Determining the precise atomic abundance of ^{107}Pd in a nuclear waste sample using the isotope dilution technique and multi-collector (MC)-ICP-MS** Majd Shmeit, Marina Faure, Celine Gautier and Hélène Isnard *Université Paris-Saclay, Service de Physico-Chimie, Gif-Sur-Yvette, France*
11:30- 11:50	**O-29**	**Re-determination of the absolute $^{13}C/^{12}C$ isotope ratio for Vienna Peedee Belemnite (VPDB) isotope-delta scale** Dmitriy Malinovskiy,[1] Philip J.H. Dunn,[1] Nives Ogrinc,[2] Doris Potočnik,[2] Lukas Flierl,[3] Olaf Rienitz,[3] Dipayan Paul,[4] and Harro A. J. Meijer,[4] Heidi Goenaga-Infante[1] [1]*National Measurement Laboratory, LGC Limited, Teddington, UK* [2] *Department of Environmental Sciences, Jožef Stefan Institute, Ljubljana, Slovenia* [3]*Physikalisch-Technische Bundesanstalt, Braunschweig, Germany* [4]*Centre for Isotope Research (CIO), University of Groningen, Groningen, Netherlands*
11:50- 12:10	**O-30**	**Direct determination of radium-226 and radium-228 in groundwater with the NexION 5000 multi-quadrupole ICP-MS** Michael Petrich, *(PerkinElmer GmbH)* and Karl Andreas Jensen *(Norwegian University of Life Sciences NMBU, Centre for Environmental Radioactivity CERAD)*
12:10- 12:30	**O-31**	**Environmental reconstruction studies through the elemental analysis of honey samples with radiocarbon based age determination** Zsófi Sajtos[a], Ágota Zsófia Ragyák[a], Tamás Varga[b], Zita Gajdos[a], A.J. Timothy Jull[b,c,d], Zsuzsa Lisztes-Szabó[b], Mihály Molnár[b], Gábor Bellér[e], Edina Baranyai[a] [a] *Environmental Analytical Research Group, Department of Inorganic and Analytical Chemistry, Faculty of Science and Technology, University of Debrecen, Hungary* [b] *International Radiocarbon AMS Competence and Training (INTERACT) Center, Institute for Nuclear Research, Debrecen, Hungary* [c] *Department of Geosciences, University of Arizona, Tucson, AZ, USA* [d]*University of Arizona AMS Laboratory, Tucson, USA* [e] *Department of Environmental Engineering, University of Debrecen, Hungary*
12:30- 13:30		**Lunch**

Session V: Vendor contributions

Chair: Dorota Bartczak

Time	Abstr.	
13:30- 13:50	**O-32**	**What to expect from modern high-temperature microwave acid digestion** Markus Michaelis and Christian Trampitsch, *Anton Paar GmbH, Graz, Austria*
13:50- 14:10	**O-33**	**Overview of a “total workflow” approach to sample prep and why it matters** Laila Kvalheim[1] and Gianpaolo Rota[2], *[1]HolgerHartmann, Langhus, Norway* *[2]Milestone, Sorisole, Italy*
14:10- 14:30	**O-34**	**Analysis of line rich matrices – Challenges and possible solutions** Petar Ivanov, Olaf Schulz and Marcus Gantenberg *Spectro Analytical Instruments GmbH, Kleve, Germany*
14:30- 14:50	**O-35**	**Automated high throughput analysis of large batches of mining discovery samples by LA-ICP-MS** Daniel Fliegel[3], Ruth Merrifield[1] and Rob Hutchinson.[2] *[1] PerkinElmer Inc., Woodbridge, ON, Canada* *[2] Elemental Scientific Europe Ltd., Huntingdon, UK* *[3] PerkinElmer Norway AS, Oslo, Norway*
14:50- 15:10	**O-36**	**Controlling sample washout** Thomas J. Kozikowski, Mike L. Booth, Autumn Phillips *Inorganic Ventures, Christiansburg, VA 24136 USA*
15:10- 15:30	**O-37**	**Automated analysis of low-to-high matrix environmental samples using ICP-MS with autodilution system** Andrew Brotherhood *Agilent Technologies, UK*

Short Course Session III

15:45- 17:30	**C1–C5**	**C1** Andreas Limbeck and Lukas Brunnbauer **Sample classification based on elemental fingerprinting: Introduction and practical guideline**	**C2** Dorota Bartczak **Engineered nanomaterials: Reference methods, reference materials and documentary standards in support of current regulations**	**C3** Jörg Feldmann and Viktoria Müller **Short course on fluorine detection with ICP-MS and ESI-MS and applications**	**C4** Walter Gössler **Arsenic speciation analysis with ICP-MS as element-selective detector**	**C5** Lana Abou-Zeid **Isotopic analysis using ICP-MS: Fundamentals and applications**
17:30- 18:00		**Coffee break, exhibition and poster viewing**				

Short Course Session IV

Time	Abstr.				
18:00-19:45	**D1–D4**	**D1** Maria Montes Bayón **ICP-MS based analytical strategies to track nanoparticles in biological systems**	**D2** Carsten Engelhard **Nanomaterials characterization by ICP-MS in single particle mode and related techniques**	**D3** Karl Andreas Jensen **Practical approaches to use reaction gases in ICP-MS triple quadrupole.**	**D4** Gary Hieftje **Make your lecture count!**
From 19:00		**Dinner**			

Wednesday, June 12, 2024

Session VI: Laser ablation applications

Chair: Vincent Motto-Ros

08:00-08:30 **O-38** **The need to meet: Mutual benefits for geochemists and chemists when discussing (LA)-ICP-MS/MS**
Thomas Zack[1,2] and Sarah Gilbert[2]
[1]*Department of Earth Sciences, University of Gothenburg, Sweden*
[2]*Department of Earth Sciences, University of Adelaide, Australia*

08:30-09:00 **O-39** **Multiscale elemental analysis of materials using laser-assisted spectrochemistry**
Andreas Limbeck, David Ken Gibbs, Maximilian Podsednik, Jakob Willner and Lukas Brunnbauer
TU Wien, Institute of Chemical Technologies and Analytics, Vienna, Austria

09:00-09:30 **O-40** **Alternative approaches to calibration in LA-ICP-MS**
Martin Šala
National Institute of Chemistry, Ljubljana, Slovenia

09:30-09:50 **O-41** **LA-ICP-MS for critical raw materials characterization: Exploring the potential of secondary resources**
Debora Foppiano, Sarina Bao, Casper Van der Eijk, Einar Jonsson and Anna Nordborg
SINTEF Industry, Trondheim, Norway

09:50-10:10 **O-42** **An approach to measure the number concentration and particle size distribution of microplastics using LA-sp-ICP-MS**
Lukas Brunnbauer, Laura Kronlachner, Elias Foisner and Andreas Limbeck
TU Wien, Institute of Chemical Technologies and Analytics, Vienna, Austria

10:10-10:40 **Coffee break, exhibition and poster viewing**
Chair: Martin Šala

10:40-11:00 **O-43** **Pseudo-bulk elemental analysis of pegmatite lithium ores by LA-ICP-MS**
Andrew M. Zipkin and Gideon Bartov
Eurofins EAG Laboratories, Liverpool, New York, USA

Time	Abstr.	
11:00-11:20	**O-44**	**Method development and acquisition parameter optimization for single pulse resolved quadrupole LA-ICP-MS multielement analysis** Jakob Willner, Lukas Brunnbauer, David Gibbs, Maximilian Podsednik and Andreas Limbeck *TU Wien, Institute of Chemical Technologies and Analytics, Vienna, Austria*
11:20-11:40	**O-45**	**Impact of histological staining on the analysis of tissue thin sections using laser ablation-inductively coupled plasma-mass spectrometry** Christine Verlemann[1], L. Schlautmann[1], M. Sperling[1], A. Radbruch[2], H. Richter[3], A. Jeibmann[4] and U. Karst[1] *1University of Münster, Institute of Inorganic and Analytical Chemistry, Germany* *[2]University Hospital Bonn, Clinic for Neuroradiology, Bonn, Germany* *[3]University of Zurich, Diagnostic Imaging Research Unit, Department of Clinical Diagnostics and Services, Vetsuisse Faculty, Zurich, Switzerland* *[4]University Hospital Münster, Institute of Neuropathology, Münster Germany*

Session VII: Bio-imaging and single cell/particle analysis

Chair: Andreas Limbeck

Time	Abstr.	
11:50-12:20	**O-46**	**Single cell ICP-MS analysis: expanding biomedical applications** Maria Montes-Bayón, L. Gutierrez Romero, C. López Portugués, P. Díez García and M. Corte-Rodríguez *Department of Physical and Analytical Chemistry, University of Oviedo, Spain* *Health Research Institute of the Principality of Asturias (ISPA). Avda. Hospital Universitario s/n, 33011, Oviedo, Spain*
12:20-12:40	**O-47**	**Molecular imaging utilizing laser ablation-atmospheric pressure chemical ionization-time of flight-mass spectrometry** Johannes Schmeinck, R. Brockington, L. Hansner, A. Kondrateva, T. Volkery and U. Karst *University of Münster, Institute of Inorganic and Analytical Chemistry, Germany*
12:40-13:00	**O-48**	**Multimodal bioimaging of intracerebral hemorrhage by use of isotopically enriched blood in a mouse model by means of LA-ICP-MS and MALDI-MSI** Peter Niehaus[1], Raphael M. Buzzi[2], Kevin Akaret[3], Michael Hugelshofer[3], Dominik J. Schaer[2] and Uwe Karst[1] *[1]Institute of Inorganic and Analytical Chemistry, University of Münster, Germany* *[2]Division of Internal Medicine, Universitätsspital and University of Zürich, Switzerland* *[3]Clinical Neuroscience Center, Universitätsspital und University of Zürich, Switzerland*
13:00-14:00		**Lunch**

Session VII continues: Bio-imaging and single cell/particle analysis

Chair: Maria Montes-Bayón

Time	Abstr.	
14:00-14:20	**O-49**	**Development and characterization of nanoparticle-embedded inks for counterfeit detection using laser ablation single particle ICP-MS** Tim Steinwachs[1], S. Baumann[2], T. Hirsch[2] and U. Karst[1] *[1]University of Münster, Institute of Inorganic and Analytical Chemistry, Germany* *[2]University of Regensburg, Institute of Analytical Chemistry, Chemo- and Biosensors, Regensburg, Germany*

Time	Abstr.	
14:20- 14:40	**O-50**	**Single cell – inductively coupled plasma – mass spectrometry (SC-ICP-MS): A powerful tool to study metals during host-pathogen cell interactions** Claire Davison[1,2], Jordan Pascoe[2], Melanie Bailey[1], Dany JV Beste[2] and Mónica Felipe-Sotelo[1]. *[1]School of Chemistry and Chemical Engineering, Faculty of Engineering and Physical Sciences, University of Surrey, Guildford, UK* *[2]Department of Microbial Science, Faculty of Health and Medical Sciences, University of Surrey, Guildford, UK*
14:40- 15:00	**O-51**	**Multi-element/isotope single-cell analysis by ICP-time of flight (TOF)-MS** Xiangwei Tian[1], Yongguang Yin[1], Yong Cai[2], Guibin Jiang[1] *[1]Research Center for Eco-Environmental Sciences, Chinese Academy of Sciences, Beijing 100085, China* *[2]Department of Chemistry and Biochemistry, Florida International University, Miami, Florida 33199, United States*
15:00- 15:20	**O-52**	**Single particle TOF-ICP-MS for the determination of atmospheric particles** Yannick Tardif, Katia Iatariene, Houssame-Eddine Ahabchane, Madjid Hadioui, Patrick Hayes and Kevin J. Wilkinson *Biophysical Environmental Chemistry, University of Montreal, QC, Canada*
15:20- 15:40	**O-53**	**Characterisation of small microplastics by spICP-MS: Dynamic mass flow method for the SI traceable quantification of number-concentration** David Ojeda, A. Sikora, D. Bartczak and H. Goenaga-Infante *LGC National Measurement Laboratory, Teddington, Middlesex, UK*
15:40- 16:00	**O-54**	**A further insight into the wider implementation of the dynamic mass flow approach for the accurate determination of particle number concentration** Armando Sánchez-Cachero, David Ojeda, Kharmen Billimoria, Dorota Bartczak, and Heidi Goenaga-Infante *LGC National Measurement Laboratory, Teddington, Middlesex, UK*
16:00- 16:20	**O-55**	**Advantages of single-cell ICP MS to monitor the metal uptake by green algae** Julia Dressler[1], Muhammad Younas[2], Alexander Köhrer[1], Matthias Elinkmann[1], Derrick Quarles Jr[3], Michael Hippler[2] and Uwe Karst[1] *[1]University of Muenster, Germany* *[2]University of Muenster, Institute of Biology and Biotechnology of Plants, Germany* *[3]Elemental Scientific, Atlanta, GA & Omaha, NE, USA*
16:40		**Departure for the excursion to the Briksdal Glacier** **with a conference outdoor dinner**

Thursday, June 13, 2024

Session VIII: Applications of plasma spectrochemistry in food, material and environmental sciences

Chair: Yngvar Thomassen

Time	Abstr.	
08:30-09:00	**O-56**	**Strategies for the reliable quantification of mass and number concentrations of nanomaterials in food: The power of a multimethod approach centered around ICP-MS** Dorota Bartczak, *LGC National Measurement Laboratory, Teddington, Middlesex, UK*
09:00-09:20	**O-57**	**Online elemental analysis in hydrometallurgical production and recycling processes of lithium-ion battery metals using micro-discharge optical emission spectroscopy** Toni Laurila and Aappo Roos *Sensmet Ltd, Finland*
09:20-09:40	**O-58**	**Challenges in the determination of toxic elements in food additives** Daniela Montalvo Grijalva[1], Céleste Vansnick[1], Noa Olluyn[1], Iris Wyns[1], Jan Mast[1], Nadia Waegeneers[2] and Karlien Cheyns[1] *[1]Sciensano, Unit trace elements, Tervuren, Belgium,* *[2]Sciensano, Unit Risk and Health Impact Assessment, Brussels, Belgium*
09:40-10:10		**Coffee break** Chair: Stefan Stürup
10:10-10:30	**O-59**	**Investigating element concentrations in water samples from the West Indian Ocean** Michaela Zeiner[a], Wolfgang Kandler[b], and Heidelore Fiedler[a] *[a]Örebro University, School of Science and Technology, MTM Research Centre, Örebro, Sweden* *[b]University of Natural Resources and Life Sciences, Vienna, Department of Agrobiotechnology, IFA-Tulln, Institute of Bioanalytics and Agro-Metabolomics, Tulln, Austria*
10:30-10:50	**O-60**	**Accurate arsenic determination in pyrolysis oils using a total sample introduction system in combination with ICP-MS/MS** Raquel Sánchez[a], Mar Todolí,[a] Rebeca Pérez,[a] Thomas Coquet,[b] Marion Lacoue-Nègre [b] and Fabien Chainet[b] *[a] Department of Analytical Chemistry, Nutrition and Food Sciences, Alicante, Spain* *[b] IFP Energies nouvelles, Rond-point de l'échangeur de Solaize, Solaize, France*
10:50-11:10	**O-61**	**Use of ICPMS in official food and feed control – experiences from the European Union reference laboratory (EURL-MN)** Jens J. Sloth, Katrin Löschner, Lucas Givelet and Heidi Amlund *National Food Institute, Technical University of Denmark, DK-2800 Kgs. Lyngby, Denmark and the European Union reference laboratory for metals and nitrogenous compounds (EURL-MN)*
11:10-11:30	**O-62**	**Plasma techniques for the analysis of lithium ion batteries** Dennis Kessen[a], Tobias Brake[a], Marc Vahnstiege[a], Malina Hellinga, Kai Brüning[a], Till Kröger[a], Martin Winter[a,b], Sascha Nowak[a] and Simon Wiemers-Meyer[a] *[a] MEET Battery Research Center, Münster, Germany* *[b] Helmholtz-Institute Münster, Forschungszentrum Jülich GmbH, Münster, Germany*
11:30		**Closing remarks and farewell**
12:00		**Lunch**

Poster Presentations

Sunday, June 9 - Wednesday, June 12, 2024

Abstr.

P-1 **Speciation analysis of Cr(III) and Cr(VI) using µLC-ICP-MS**
Jelle Verdonck[1], Katrien Poels[1], Jeroen Vanoirbeek[1], Radu Corneliu Duca[1,2], Lode Godderis[1,3] and Erk Smolders[4]
[1]Centre for Environment and Health, Department of Public Health and Primary Care, KU Leuven (University of Leuven), Leuven, Belgium
[2]Environmental Department Health Protection, Laboratoire National de Santé (LNS), Dudelange, Luxembourg
[3]IDEWE, External Service for Prevention and Protection at Work, Heverlee, Belgium
[4] Division of Soil and Water Management, Department of Earth and Environmental Sciences, KU Leuven (University of Leuven), Leuven, Belgium

P-2 **The presence and behavior of titanium and silver nanoparticles in a water treatment plant and wastewater treatment plant located in the Llobregat River (Barcelona, Spain)**
A. Díaz[1], E. Guiot[1], I. Perez[1], E. Peña-Vázquez[2], M.C. Barciela-Alonso[2]. M. Aboal-Somoza[2] and P. Bermejo-Barrera[2]
[1] Aigües de Barcelona (Agbar). Laboratory. General Batet 5-7, Barcelona, Spain
[2] Trace Element, Spectroscopy and Speciation Group (GETEE), Instituto de Materiais (iMATUS), Faculty of Chemistry, University of Santiago de Compostela, Spain

P-3 **Improving image quality and pixel resolution via laser pulse dosage increase for fast imaging of large areas with LIBS**
Jakob Willner[1], Lukas Brunnbauer[1], David Gibbs[1], Maximilian Podsednik[1] and Andreas Limbeck[1]
[1]TU Wien, Institute of Chemical Technologies and Analytics, Vienna, Austria

P-4 **Thin water films transport compounds from the leaf surface into the plant tissue - Laser Ablation ICP-MS reveals the pathways**
Max Frank[*1], Francesca Siracusa[2], Daniel P. Persson[1], Rajmund Mokso[2] and Søren Husted[1]
[1] Department of Plant and Environmental Sciences (PLEN), University of Copenhagen (KU), Denmark,
[2] Department of Physics, Technical University of Denmark (DTU), Denmark.

P-5 **Rapid microwave-assisted acid digestion of heavy petroleum products for trace metal analysis by ICP-OES and ICP-MS**
Markus Michaelis[1] and Glenn Müller[2],
[1]Anton Paar GmbH, Graz, Austria
[2]Dipl.ing. Houm AS, Oslo, Norway

P-6 **Li-ion battery materials digested for ICP-OES analysis**
Markus Michaelis[1], Gisela Fauler[1] and Glenn Müller[2]
[1]Anton Paar GmbH, Graz, Austria
[2] Dipl.Ing. Houm AS, Oslo, Norway

P-7 **Ever lower limits challenge ICP-MS food analysis - How microwave acid digestion helps**
Klaus Pichler-Jöbstl and Markus Michaelis
Anton Paar GmbH, Graz, Austria

P-8 **Acid digestion for PGM recycling analysis with ICP-MS**
Markus Michaelis and Peter Kettisch
Anton Paar GmbH, Graz, Austria

Abstr.

P-9 **Acid digestion of high purity PGMs including rhodium, iridium and ruthenium**
Markus Michaelis and Peter Kettisch
Anton Paar GmbH, Graz, Austria

P-10 **Hyphenating HPLC and ICP-MS: The case of polysulfide anions**
Aleksei Sadykov[a], Martin Winter[a,b], Simon Wiemers-Meyer[a] and Sascha Nowak[a]
[a]*University of Münster, MEET Battery Research Center, Münster, Germany*
[b]*Helmholtz-Institute Münster, IEK-12, Forschungszentrum Jülich GmbH, Münster*

P-11 **Combining full spectrum read-out and innovative automation capabilities for robust and fast analysis using ICP-OES**
Mikael Axelsson, Sukanya Sengupta and Daniel Kutscher
ThermoFisher, Stockhom, Sweden

P-12 **Accurate and reliable multielement analysis of alternative protein foods using ICP-MS**
Mikael Axelsson, Sukanya Sengupta and Daniel Kutscher
ThermoFisher, Stockholm, Sweden

P-13 **Nano-hydroxyapatite as a novel phosphorus fertilizer**
Francesco Minutello, Daniel P. Persson and Søren Husted
University of Copenhagen, Department of Plant and Environmental Sciences, Frederiksberg C, Denmark

P-14 **Optimization of a method for determination of stable boron isotope ratios in various aqueous matrices using multi-collector inductively-coupled plasma mass spectrometry**
Julianne Hem[1] and Christian Schöpke[1,2]
[1]*Norwegian University of Life Sciences (NMBU), Ås, Norway*
[2]*Institute for Energy Technology (IFE), Kjeller, Norway*

P-15 **Copper speciation in human lung tissue: Comparative analysis between squamous-cell carcinoma and healthy lung tissue using HPLC-ICP-MS**
Katarina Marković[1], Aleksandar Stojsavljević[3], Jernej Šribar[4], Igor Križaj[4,2], Radmila Milačič[1,2] and Janez Ščančar[1,2]
[1]*Department of Environmental Sciences, Jožef Stefan Institute, Ljubljana, Slovenia*
[2]*Jožef Stefan International Postgraduate School, Ljubljana, Slovenia*
[3]*Innovation Center, Faculty of Chemistry, University of Belgrade, Belgrade, Serbia*
[4]*Department of Molecular and Biomedical Sciences, Jožef Stefan Institute, Ljubljana, Slovenia*

P-16 **The study of lactoferrin-ruthenium complexes: Insight into the binding mechanism**
Tetiana Dyrda-Terniuk[1] and Paweł Pomastowski[1,2]
[1] *Centre for Modern Interdisciplinary Technologies, Nicolaus Copernicus University in Toruń, Poland*
[2] *Department of Inorganic and Coordination Chemistry, Faculty of Chemistry, Nicolaus Copernicus University in Toruń*

P-17 **The impact of production processes on selected heavy metals distribution in dairy products of Kuyavian-Pomerian Voivodeship, Poland**
Oleksandra Pryshchepa[1] and Paweł Piotr Pomastowski[1,2]
[1]*Centre for Modern Interdisciplinary Technologies Nicolaus Copernicus University in Torun, Torun, Poland*
[2]*Department of Inorganic and Coordination Chemistry, Faculty of Chemistry, Nicolaus Copernicus University in Torun, Poland*

Abstr.

P-18 **Trace element analysis of pegmatite lithium ores by GD-MS**
Gideon Bartov and Andrew M. Zipkin
Eurofins EAG Laboratories, Liverpool NY, USA

P-19 **Advancing stable and long-lived radiogenic isotope ratio measurements of environmental pollutants**
Tea Zuliani and more (see abstract),
Jožef Stefan Institute, Department of Environmental Sciences, Ljubljana, Slovenia,

P-20 **Determination of critical raw materials in micromobility e-vehicles by ICP-OES**
Riikka Turkki, Siiri Perämäki and Ari Väisänen
Department of Chemistry, University of Jyväskylä, Finland

P-21 **Elemental analysis of mango flesh and peel by ICP-MS**
Michaela Zeiner[1], Ema Mihalić[2], Iva Juranović Cindrić[2], Ivan Nemet[2] and Heidelore Fiedler1
[1]Örebro University, School of Science and Technology, Man-Technology-Environment Research Centre, Örebro, Sweden
[2]Sveučilište u Zagrebu Prirodoslovno-matematički fakultet, Kemijski odsjek, Zavod za analitičku kemiju, Zagreb, Croatia

P-22 **Analysis of plant material from phytoremediation processes of soils contaminated with lithium ion battery materials using plasma-based methods**
Julius Buchmann[a], Martin Winter[a,b] Simon Wiemers-Meyer[a] and Sascha Nowak[a]
[a]University of Münster, Münster Electrochemical Energy Technology (MEET), Germany
[b]Helmholtz-Institute Münster (HIMS), IEK-12, Forschungszentrum Jülich, Münster

P-23 **Recovery and ICP-OES analyses for selected metals from electronic waste and rechargeable batteries**
Antti Tiihonen[1], Jutta Koskinen[1], Siiri Perämäki[1], Janne Frimodig[1], Jimi Siljanto[1], Joona Rajahalme[2], Sylva Larsson[2], Elmeri Lahtinen[2], Jannatul Rumky[1], Joni Niskanen[2], Roshan Budhathoki[2], Manu Lahtinen[1], Matti Haukka[1] and Ari Väisänen[1]
[1] Department of Chemistry, P.O. Box 35 FIN-40014 University of Jyväskylä, Finland
[2] Formerly University of Jyväskylä, currently affiliated in various industrial sectors

P-24 **Understanding water bodies in and around Romerike region in Norway through strontium isotope analysis using MC-ICP-MS**
Vani N. Devegowda and Christian Schöpke
Institute for Energy Technology (IFE), Kjeller, Norway

P-25 **Analysis of petroleum products according to ASTM method D8110-17**
Ewa Pruszkowski [1], Chady Stephan [1] and Fliegel Daniel[2]
[1]PerkinElmer Inc., USA and Canada
[2]PerkinElmer Norway AS, Oslo, Norway

P-26 **The analysis of drinking water using universal collision-reaction gas technology on the NexION 1100 ICP-MS**
Pritesh Patel[1] and Zuo, Minyu[2]
[1]PerkinElmer Inc., USA and Canada
[2]PerkinElmer, Stockholm, Sweden

P-27 **Enhancing trace element determination in seawater using NexION ICP-MS systems**
Liyan Xing[1], Ewa Pruszkowski[1], Chady Stephan[1], Aaron Hineman[1] and Daniel Fliegel[2]
[1]PerkinElmer U.S. LLC
[2]PerkinElmer Norway AS, Oslo, Norway

Abstr.

P-28 **Investigation of the charge state of lithium ion battery active materials by means of single particle ICP-OES**
Marc Vahnstiege[1], Alexandros Tsoufios[1], Martin Winter[1,2], Sascha Nowak[1] and Simon Wiemers-Meyer[1]
[1] *University of Münster, MEET Battery Research Center, Münster, Germany*
[2] *Helmholtz-Institute Münster, IEK-12, FZ Jülich, Münster, Germany*

P-29 **Determination of elements and element species in size resolved urban aerosol samples by HPLC-ICP-MS/MS**
Stefan Tanda[1], Balázs Berlinger[2] and Walter Gössler[1]
[1] *Department of Chemistry, University of Graz, Graz, Austria*
[2] *Department of Animal Hygiene, Herd Health and Mobile Clinic, University of Veterinary Medicine, Budapest, Hungary*

P-30 **Elemental and isotopic analysis of aged honeys – A North American perspective on unique sweeteners**
Ágota Zsófia Ragyák, Zsófi Sajtos and more (see abstract
[a] *Environmental Analytical Research Group, Department of Inorganic and Analytical Chemistry, Faculty of Science and Technology, University of Debrecen, Hungary*

P-31 **Quantitative imaging of a lunar meteorite sample by laser-induced breakdown spectroscopy trained on energy dispersive spectroscopy data**
Daniel Holub[1,2], Timon Schild[3,4], Dennis Harries[3,5], Pavel Pořízka[2,6], Jozef Kaiser[1,2,6]
[1] *Faculty of Mechanical Engineering, Brno University of Technology, Brno, Czech Republic*
[2] *CEITEC Brno University of Technology, Brno, Czech Republic*
[3] *European Space Resources Innovation Centre, Esch-sur-Alzette, Luxembourg*
[4] *University of Luxembourg, Esch-sur-Alzette, Luxembourg*
[5] *Luxembourg Institute of Science and Technology, Esch-sur-Alzette, Luxembourg*
[6] *Lightigo s.r.o., Renneská třída 329/13, 639 00 Brno – Štýřice, Czech Republic*

P-32 **Dental ankylosis' developmental stages determination by correlative imaging and data fusion**
Anna Konečná[1], Daniel Holub[1,2], Jan Štembírek[3,4], Pavel Hurník[4,5,6], Marcela Buchtová[4,7], Pavel Pořízka[1,2,8], Jozef Kaiser[1,2,8]
[1] *CEITEC Brno University of Technology, Purkyňova 123, 612 00, Brno, Czech Republic*
[2] *Faculty of Mechanical Engineering, Brno University of Technology, Brno, Czech Republic*
[3] *Department of Oral and Maxillofacial Surgery, University Hospital Ostrava, Ostrava-Poruba, Czech Republic*
[4] *Laboratory of Molecular Morphogenesis, Institute of Animal Physiology and Genetics, Czech Academy of Sciences, Veveří 967/97, 602 00 Brno, Czech Republic*
[5] *Institute of Molecular and Clinical Pathology and Medical Genetics, University Hospital Ostrava, Ostrava-Poruba, Czech Republic*
[6] *Institute of Molecular and Clinical Pathology and Medical Genetics, Faculty of Medicine University of Ostrava, Syllabova 19, 703 00 Ostrava, Czech Republic*
[7] *Department of Experimental Biology, Masaryk University, Brno, Czech Republic*
[8] *Lightigo s.r.o., Renneská třída 329/13, 639 00 Brno – Štýřice, Czech Republic*

12 Oral Abstracts

(O-1)

Advanced techniques in plasma spectrochemistry: Origins and future

Gary M. Hieftje

Department of Chemistry, Indiana University, Bloomington, IN 47405, USA

E-mail: hieftje@iu.edu

Plasma Spectrochemistry, like most analytical methods, generally advances by means of an evolutionary process: incremental innovations and better understanding result in progressive improvements in performance, user-friendliness, and utility. Conveniently, this sort of process lends itself to realistic extrapolation, fostering further innovation and simplifying such practical matters as market prediction and the preparation of "future-oriented" lectures. However, a few advances seem disruptive; they were not earlier anticipated, appear to have come from nowhere, yet have a major impact. Such developments would seem to be impossible to predict. Yet, their origins can be traced, albeit often to unrelated events, external pressures, and unanticipated needs [1]. In this lecture, the origins of several spectrochemical techniques will be examined to identify factors and earlier occurrences that led to their current prominence; in turn, modern needs and pressures (e.g. growing populations, energy shortages, climate change) will be combined with several earlier and recent developments (e.g., artificial intelligence, quantum entanglement and computing, optical modulation) to suggest advanced spectroscopic techniques that are likely to grow in importance and others that might lie on the horizon.

[1] Mauro F. Guillén, *"2030: How Today's Biggest Trends Will Collide and Reshape the Future of Everything"*, St. Martin's Press, New York, 2020.

(O-2)

Acoustic fields to shape, move, and gate ion beams for mass spectrometry

Jacob T. Shelley,[1] Yi You,[2] Julia L. Danischewski,[1] Lauren S. Bauer,[1] and Jens Riedel[2]

[1] *Department of Chemistry and Chemical Biology, Rensselaer Polytechnic Institute, Troy, NY 12180 USA*
[2] *Department of Instrumental Analytics, Federal Institute for Materials Research and Testing (BAM), Berlin 12489 Germany*

Approaches to control the motion and direction of ionized atoms and molecules are an essential aspect of ion-based analytical methods, such as mass spectrometry (MS) and ion-mobility spectrometry (IMS). Because these species are inherently charged, most ion-manipulation approaches rely upon electrostatic and/or Lorentz forces in electric fields and magnetic fields, respectively. The ability to produce intact gaseous ions for MS and IMS is often performed at atmospheric-pressure (AP) due ease of sample introduction, high ionization efficiencies, and minimal fragmentation. However, diffusion and electrostatic repulsion between ions hinders the transport of gaseous ions into the lower-pressure environment of the analyzers. Conventional ion optics, that use electric or magnetic fields, can guide ions at AP, but require high field strengths to overcome the dominating aerodynamic effects.

Here, we describe a remarkable phenomenon whereby low-power acoustic fields are used to move, shape, gate, and separate beams of gaseous ions at atmospheric pressure. We refer to this approach as Acoustic Ion Manipulation Spectrometry (AIMS). Gaseous ions formed by AP electrical plasmas and other ionization approaches are directed towards, and separated by, the presence of the acoustic field. To better understand the phenomenon, an ion-detector array provided a measure of bulk ion movement, while mass spectrometry (MS) offered chemical-specific information. As one example of an AIMS setup, a standing acoustic wave was formed with two ultrasonic speakers and placed between an ionization source and ion detector. Ion beams are deflected away from unstable pressure regions (i.e. antinodes) into the pressure-stable nodal areas. Shadowgraphy revealed that the neutral gas stream continued through an antinode and, thus, ions were separated from the transport gas. Interestingly, both positive and negative ions follow this same trajectory indicating that charge, but not polarity, is a driving factor in AIM. Specific examples of ion focusing, gating, and separation (based on ion size) will be shown. Ion gating efficiencies of more than 99% have been achieved a single acoustic resonator. In addition to the empirical description of the AIM phenomenon, the use of complex acoustic field structures for improved ion gating and focusing will be presented. This discovery could have profound impacts in analytical ion-based spectrometries, like MS and IMS, as well as in materials processing and characterization.

(O-3)

Advancements in elemental and molecular mass spectrometry for the characterization of nanoparticles and microplastics

Carsten Engelhard[1,2], Annika Schardt[2], Johannes Schmitt[2], Cristian C. Escobar-Carranza[2], Désirée A.-M. Schütz[2] and Manuel Heinelt[2]

[1]*BAM, Bundesanstalt für Materialforschung und -prüfung, Richard-Willstätter-Straße 11, 12489 Berlin*
[2]*University of Siegen, Department of Chemistry and Biology, Adolf-Reichwein-Str. 2, D-57076 Siegen, Germany*

e-mail: carsten.engelhard@bam.de

In the first part of the presentation, the current state-of-the-art in single-particle (sp) inductively coupled plasma mass spectrometry (ICP-MS) instrumentation will be discussed. While millisecond dwell times were used in the advent of spICP-MS, the use of microsecond dwell times helped to improve nanoparticle data quality and particle size detection limits. Our latest development is an in-house built data acquisition system with nanosecond time resolution (nanoDAQ). Recording of the SEM signal by the nanoDAQ is performed with a dwell time of approximately 4 ns. A tailored method was developed to process this type of transient data, which is based on determining the temporal distance between detector events that is denoted as event gap (EG). We found that the inverse logarithm of EG is proportional to the particle size and that the number of detector events corresponding to a particle signal distribution can be used to calibrate and determine the particle number concentration (PNC) of a nanoparticle dispersion. Due to the high data acquisition frequency, a statistically significant number of data points can be obtained in 60 s or less. Successful detection of gold nanoparticles (AuNP) as small as 7.5 nm was demonstrated using a single quadrupole ICP-MS.

Microplastics (MPs) can now routinely be identified and characterized using standard methods such as Fourier transform infrared (FTIR) and Raman spectroscopy combined with optical microscopy. Additionally, pyrolysis or thermal extraction and desorption units coupled with gas chromatography-mass spectrometry (GC-MS) are used for MP analysis. While these methods are powerful, a complementary and rapid screening tool for the direct chemical characterization of MPs would be an interesting addition to the toolbox. Here, ambient desorption/ionization mass spectrometry (ADI-MS) is considered attractive because it does not require a preceding chromatography step and provides simultaneous mass spectral information over a wide mass range. Specifically, the use of ADI-MS with a home-built flowing atmospheric-pressure afterglow (FAPA) source [1-2] for the direct characterization of different analytes and plastic materials will be discussed. Various plastic materials were probed, and differentiation of different plastic types was performed with principal component analysis (PCA). With the aid of thermal desorption and a halo-shaped (h-FAPA) configuration (based on [3]) the performance could be improved. Recent results will be discussed.

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(O-4)

Quantitative nano depth profiling by means of high-energy laser desorption ionization (HELDI)

Davide Bleiner[1,2], Sharath Rameshbabu[1,2], and Janosch von Ballmos[2]

[1]University of Zurich – Dept. Chemistry, University of Zurich, Winterthurerstrasse 190, CH-8057 Zurich, Switzerland; [2]Empa – Swiss Federal Laboratories for Materials & Technology, Überlandstrasse 129, CH-8600 Switzerland

E-mail: davide.bleiner@uzh.ch

Direct nano-scale microanalysis is important for a number of materials science applications, e.g. photovoltaic functional thin films or catalysts, in order to characterize their nano-trace homogeneity and purity. This demands combining spatial resolution iand sensitivity, at a level (at the moment) beyond state-of-the-art. As dictated by counting statistics, the reduction of the spot size degrades the detection limit.

The utilization of a prototype tabletop XUV laser at $\lambda = 46.9$ nm has shown to dramatically improve the ablation efficiency [1-2]. The reduction of the wavelength is an alternative approach to the reduction of the pulse duration. XUV-laser ablation combines elemental and molecular information. 3D nano-chemical plots are produced with the aid of supervised machine learning. High (photon) energy laser desorption ionization (HELDI) is a novel microanalytical technique using XUV laser pulses to enhance and homogenize sensitivity at nano-scale, especially for light elements. While inspecting nano-structures in 3D [3-4], local heterogeneities are visible only if the instrumental variance can be discerned from compositional one. The results indicated an enhanced analytical capability for light elements imaging and the ability to discern instrumental variances (random error) from true compositional ones (heterogeneity).

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(O-5)

25 years of the dynamic reaction cell - Predictable reactions across the entire periodic table

Minyu Zuo[1], Michael Petrich[1] and Karl Andreas Jensen[2]

[1] *PerkinElmer, Svampvägen 12, 18741 Täby, Sweden*
[2] *Centre for Environmental Radioactivity – CERAD, Norwegian University of Life Sciences, Ås, Norway*

The advent of the first commercial inductively coupled plasma mass spectrometer (ICP-MS) in 1983 marked a significant milestone in analytical chemistry, promising a future free from the constraints of interference. However, decades of research have revealed the persistent challenge of both non-spectral and spectral interferences. This study delves into the innovative strategies employed by the NexION 5000 multi-quadrupole ICP-MS to confront these interferences, highlighting the pivotal role of reaction gases in their mitigation.

The ICP-MS/MS technology reinforces the trend of using chemical resolution with very reactive gases, because the focus only must be on different yields of analyte and interferents on the target mass. Possible reactions of all other ions formed in the plasma with the reaction gas are completely prevented in ICP-MS/MS. Color-coded periodic tables and a database of product ion scans serve not only as a beacon for method development in complex matrices but also as a troubleshooting compass.

The optimization of the cell settings, on the other hand, plays a minor role compared to classical ICP-MS. This makes method development much easier and allows the use of reactive gases even for short transient signals where many analytes have to be measured with one cell gas setting.

The synergy of multi-quadrupole technology and the Universal Cell within the NexION 5000 is showcased, demonstrating unparalleled efficiency in the removal of spectral interferences, thereby setting new benchmarks for Background Equivalent Concentrations (BECs) and Detection Limits (DLs).

This work affirms that while interferences in ICP-MS are an inevitable reality, the cutting-edge NexION 5000 stands as a testament to the power of technology to overcome analytical obstacles, ensuring precise and reliable results.

(O-6)

Coupling laser ablation SF-ICP-MS, Q-ICP-MS, MC-ICP-MS and ICP-OES with electron microscopy for interdisciplinary (geology-biology-archaeology) research

Tonny B. Thomsen, Benjamin D. Heredia, Sebastian N. Malkki and Nynke Keulen

The Geological Survey of Denmark and Greenland (GEUS), Copenhagen, Denmark

E-mail: tbt@geus.dk

The use of laser ablation (LA) coupled with inductively coupled plasma (ICP) mass spectrometry (MS) or optical emission spectrometry (OES) has today become a very important analytical tool in Earth and Environmental science for the determination of isotopic or elemental compositions from natural materials or products manufactured from natural materials. At GEUS this application versatility is explored further through the development of an approach where point-to-point coordination routine on µm scale is integrated to LA-based analyses using sector-field (SF) ICP-MS, quadrupole (Q) ICP-MS, or ICP-OES with measurements obtained by electron microscopy (SEM, EMPA) or traditional optical microscopy. Analysis at exact same locations obtained by the different instruments has proven its high value in several projects and an accurate coordination from, for instance, SEM to LA-ICP-MS is critical when visual identification is not possible such as for U-Pb geochronometry combined with elemental analysis measured from tiny minerals in stone axes and objects that cannot be cut and polished. Examples using this approach will be presented.

The recent addition of multi-collector (MC) ICP-MS instrumentation and the setup of a split-stream methodology in our laboratory has further offered the opportunity to expand the use of the point-to-point coordination approach. Thus, the MC-ICP-MS makes it possible to analyse for other isotopes and determine paired isotopic ratios (e.g. of Hf, Sr, Li, Pb, Mg, S, Cu, Sm-Nd, Re-Os) in addition to our routine U-(Th-)Pb dating, isotopic and elemental analyses acquired by the SF-ICP-MS, Q-ICP-MS, and ICP-OES. Simultaneous, high through-put acquisition of data from the exact same points locations, lines or curves guided by our point-to-point coordination approach thus provide more accurately correlating data (µm scale) from all analytical instruments connected concurrently. The approach makes detailed and robust correlation between data points from different instruments, facilitating advances towards a more coherent data interpretation, something that especially projects of a complex character may significantly benefit from.

To further enhance the analytical capability of the laboratory, our future target includes a femto-second LA with an integrated LIBS option, which will allow us to measure light elements, in particular. Compared to our existing nano-second LA instruments, a femto-second LA will generally provide better performance with reduced analytical uncertainties, better sensitivity and ablation yield, and potentially improve the spatial resolution of the analyses, thus contributing with a paradigm-shift on the uses of different instrumentation.

(O-7)

Advancements in elemental analysis of lithium ion batteries using ETV-ICP-OES – Challenges and opportunities

Dennis Kessen[1], Matthias Walter[1], Martin Winter[1,2], Sascha Nowak[1] and Simon Wiemers-Meyer[1],

[1] University of Münster, MEET Battery Research Center, Corrensstraße 46, 48149 Münster, Germany
[2] Helmholtz-Institute Münster, IEK-12, FZ Jülich, Corrensstraße 46, 48149 Münster, Germany

Lithium ion batteries (LIBs) are an integral part of our modern lifestyle, powering everything from consumer electronics and power tools to electric vehicles. Despite their ubiquity, LIB calendar and cycle lifetime are still compromised by several aging mechanisms that occur during storage and operation of the battery. Elemental analysis plays an important role in unraveling these aging mechanisms. Inductively coupled plasma-optical emission spectroscopy (ICP-OES) is e.g., commonly used to quantify the loss of cyclable lithium and deposited metals (Ni, Co, Mn) on the negative electrodes, which stem from material instability towards the harsh working conditions of the LIB and subsequent crosstalk phenomena.[1]

Coupling electrothermal vaporization (ETV) to ICP-OES offers distinct advantages over conventional microwave-assisted digestion approaches. By eliminating the time-consuming and labor-intensive digestion step, analysis time can be reduced with less risk of contamination, a potential gain in sensitivity and the possibility to analyze hard-to-digest materials.[2] In addition, temperature-controlled vaporization may provide novel insights into the composition of the LIB electrode interphases, comprising a mixture of various inorganic lithium salts as well as organic carbonates, phosphates, oligo- and polymers. This presentation provides a brief overview on method development and the benefits using ETV-ICP-OES for the elemental analysis of LIBs and addresses the challenges encountered with the nature of aged electrodes.

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(O-8)

Plasma mass spectrometry in speciation analysis - present and future

Joanna Szpunar

IPREM, UMR5254 CNRS-UPPA, Hélioparc, 2, Av. Angot, 64053 Pau, France

E-mail: joanna.szpunar@univ-pau.fr

Interest in elucidating the mechanisms governing the role of trace metals in biology has driven the search for suitable analytical methods for elemental species analysis (speciation) since the 1990s. Hyphenated (coupled) techniques, which combine a separation technique (such as chromatography or electrophoresis) and element-specific detection - typically using inductively coupled plasma mass spectrometry (ICP MS) - have revolutionized the exploration of the world of biological metal species and have emerged as the principal analytical approach for speciation analysis. These techniques offer high selectivity, facilitated by the separation technique, as well as sensitivity reaching down to the lower picogram levels provided by plasma mass spectrometry.

However, elemental speciation analysis still faces numerous challenges, particularly in the analysis of non-metal species, the speciation of emerging inorganic contaminants (such as technologically critical elements), and the characterization of high molecular weight (HMW) and water-insoluble species. The methods should also be capable of covering large populations of species of one or several elements, which require both high sensitivity and selectivity. Additionally, *de novo* identification of unknown species and their standardless quantification pose significant hurdles, asking for innovative approaches and advanced instrumentation. Furthermore, integrating speciation information with the biological role of elements, a field referred to as metallomics, adds another layer of complexity; it requires interdisciplinary collaboration and sophisticated data analysis techniques to elucidate the relationship between metal speciation and biological function. Addressing these challenges is essential for advancing our understanding of elemental speciation and its implications for human health and the environment.

The lecture, based on examples from the author's laboratory, will address the role played in the elemental speciation analysis by plasma spectrometry complemented by the state-of-the-art electrospray ionization high resolution accurate mass (HRAM) mass spectrometry that makes possible targeting the entire molecules and providing an insight into the structure of unknown species.

(O-9)

A closer look to the elemental world of fungi with HPLC coupled to ICP-MS

Walter Gössler

University of Graz, Austria

Fungi have an important position in ecosystems as symbionts, saprotrophs, and parasites. The roles of fungi involve element cycling, rock and mineral transformations, bioweathering, fungal-clay interactions, and metal-fungal interactions. Therefore, fungi can liberate elements from soil, effectively transform halogens, metals, metalloids, and organometallic compounds by reduction, methylation, and dealkylation. These processes are very important for our ecosystems since transformations of metal(loid)s modify their mobility and toxicity.

As it is really difficult to remove soil or rotted wood from the mycelium fruit bodies are mainly researched for the transformed elements from soil. Moreover fruit-bodies serve as important nutrient source for parasites, wild animals, and also humans. It is not commonly known that fruit bodies may accumulate remarkable concentrations of (trace) elements. The well-known *King Bolete* (Boletus edulis) is known for its ability to accumulate the essential trace element selenium but on the other hand this fungus also accumulates significant amounts of mercury. This heavy metal is well known for its toxic properties. *The Fly Agaric* (Amanita muscaria) is a selective vanadium accumulator. Several 100s of mg V/kg dry mass have been determined during our studies [1]. *Thelephora penicillata* was found to be a cadmium accumulating mushroom (more than 1000 mg Cd/kg). Additionally, this mushroom accumulated high concentrations of arsenic, too [2]. The *Violet Crown Cup* (Sarcosphaera coronaria), a mushroom considered edible in previous days, has been reported as an arsenic accumulator. We have determined close to 1 % of arsenic on a dry mass basis [2]. Such concentrations are hardly ever reported for any natural living organism. The edible Ink Stain Bolete (Cyanoboletus pulverulentus) is also capable to accumulate up to 1300 mg As/kg.

As not all arsenic compounds have the same toxic properties it is necessary to determine the arsenic speciation. Our research provided new results with respect to the arsenic speciation in mushrooms. During our work we discovered several new arsenicals for the first time in a living organism [3].

The presentation covers analytical aspects as well as the results for the total element determinations and the elemental speciation.

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(O-10)

A new approach to speciation stabilization – Species separation at the sampling site

Beata Krasnodębska-Ostręga, Joanna Kowalska, Katarzyna Kińska, and Monika Sadowska

Faculty of Chemistry, University of Warsaw, Poland
E-mail: bekras@chem.uw.edu.pl

Speciation analysis is subject to errors resulting from the very fact of sampling, when the primary equilibrium is altered due to many transformations. It is quite difficult to prevent changes occurring between individual chemical forms of analytes during sample collection, but also during transport and storage in the laboratory. Importantly, sample preservation itself also affects speciation [1]. These problems occur with As, Cr, Te and Tl. Tl(III) ions are reduced to Tl(I) even during fixation by the addition of DTPA [1]. In the case of As(III), Cr(III) and Te(IV), oxidation is a consequence of contact with oxygen and light, immediately after sampling from a water reservoir [1,2]. The changes can reach up to 10-22%. A solution to this problem is the new strategy to preserve speciation without chemical fixation and just after sampling. The essence of the procedure is based on selective extraction of the speciation forms from water samples using solid-phase extraction (SPE).

Such strategy is already used in the case of As and Tl [3,4]. We also have some proposals for Te and Cr, based on our own research and literature data. Such sample pretreatment of water samples, i.e. using SPE columns, allows for speciation evaluation based on ICP-MS or ICP-OES determination. During the presentation, the risks of changing speciation during sampling and sample pretreatment before the analysis will be indicated, and the possibility of using solid-phase extraction to preserve the original speciation of As, Cr, Te and Tl directly at the sampling site will be showed. Both direct and indirect speciation analysis will be discussed.

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(O-11)

Selenium speciation studies in cancer patients to evaluate the responses of biomarkers of selenium status to different selenium compounds

Christian Ward-Deitrich[1], M. Estela del Castillo Busto[1,2], Stephen O. Evans[3,4], Margaret P. Rayman[5], Michael B. Jameson[4,6] and Heidi Goenaga-Infante[1]

[1] LGC National Measurement Laboratory (NML), Queens Road, Teddington, Middlesex TW11 0LY, United Kingdom
[2] Grupo Química Analítica Aplicada (QANAP), Instituto Universitario de Medio Ambiente (IUMA), Universidade da Coruña (UDC), 15071 A Coruña, Spain
[3] Department of Biological Sciences, University of Waikato, Hamilton, New Zealand
[4] Waikato Clinical Campus, University of Auckland, Hamilton, New Zealand
[5] Department of Nutritional Sciences, University of Surrey, Guildford GU2 7XH, United Kingdom.
[6] Oncology Department, Waitako Hospital, Hamilton, New Zealand

Email: christian.ward-deitrich@lgcgroup.com

This work presents the first systematic comparison of selenium (Se) speciation in plasma from cancer patients treated orally with three Se compounds namely sodium selenite (SS); L-selenomethionine (SeMet) or Se-methylselenocysteine (MSC) at 400 µg/day for 28 days. The study aim was to investigate, how these Se species affect the plasma Se distribution, and to identify the most effective Se compound for optimal selenoprotein expression. This was achieved using methodology based on HPLC-ICP-MS after employing different sample preparation/fractionation approaches. Measurements of total Se in plasma samples collected before and after 4 weeks of treatment showed that median total Se levels increased significantly from 89.6 to 126.4 µg kg^{-1} Se ($p<0.001$), particularly when SeMet was administered (190.4 µg kg^{-1} Se). Speciation studies showed that the most critical differences between treated, and baseline samples were seen for selenoprotein P (SELENOP) and selenoalbumin (SeAlb) after administration with MSC ($p=5.8x10^{-4}$) and SeMet ($p=6.8x10^{-5}$), respectively. Notably, selenosugar-1 was detected in all low molecular-weight plasma fractions following treatment, particularly with MSC. Two different chromatographic approaches and spiking experiments demonstrated that about 45 % of that increase in SELENOP levels (to ~8.8 mg L^{-1} as protein) with SeMet is to be expected due to the non-specific incorporation of SeMet into the SELENOP affinity fraction. To the authors' knowledge, this has not been reported to date. Therefore, SELENOP is part of both the regulated (55 %) and non-regulated (45 %) Se pools after SeMet administration, whereas SS and MSC contribute preliminary to the regulated one.

Keywords: Selenium speciation, sodium selenite, seleno-L-methionine, Se-methylselenocysteine, clinical trial, ICP-MS

(O-12)

Robust sample preparation for automated speciation analysis of gadolinium-based contrast agents in tissue samples

Torben J. Maas and Uwe Karst

University of Münster, Institute of Inorganic and Analytical Chemistry, Corrensstr. 48, 48149 Münster, Germany

Since their first approval over three decades ago, gadolinium-based contrast agents (GBCAs) have become an essential medical tool for improving image contrast in magnetic resonance imaging (MRI). These agents are created using linear or macrocyclic aminopolycarboxylic acid chelating ligands to circumvent the toxicity of free Gd3+ ions. The resulting metallopharmaceuticals are highly stable and have an overall good safety profile. After intravenous administration during an MRI exam, the highly polar complexes are typically excreted unmetabolized within a few hours. However, the retention of gadolinium has been observed in diverse body tissues and linked to various safety concerns, although the mechanism behind this retention after GBCA administration remains unknown. As a result, there is great interest in obtaining species-related information about the binding form of gadolinium in these depositions. By combining anion-exchange chromatography (IC) with inductively coupled plasma-mass spectrometry (ICP-MS), speciation analysis of individual Gd compounds is achieved. The focus of this work was to develop an appropriate sample preparation method for the speciation analysis of Gd-containing tissue with robustness regarding analyte integrity and recovery.

Tissue was first homogenized using a bead mill-assisted approach. Different bead materials were evaluated concerning the release of metal ions during homogenization and subsequent species conversion of contrast agents. Transmetalation of the linear GBCA gadodiamide with transition metal ions released from ceramic beads was observed using speciation analysis with IC-ICP-MS and could be averted by using glass-based beads. An automated single-platform system allowed for rapid chromatography within two minutes and external calibration from single stock standards. After this, the next sample preparation step involved the centrifugation of tissue homogenate to obtain the water-soluble fraction as supernatant. To eliminate matrix components that interfere with speciation analysis, the supernatant was subjected to centrifugal ultrafiltration. Only filters using a membrane based on polyethersulfone showed high recoveries of both macrocyclic and linear GBCAs. Conversely, membranes based on composite regenerated cellulose showed a decreasing recovery with an increase in the contrast agent’s charge. Finally, the robustness of the sample preparation method was assessed by spiking calf’s liver as a model tissue matrix with the linear gadodiamide. The results of speciation analysis using IC-ICP-MS revealed gradual degradation of the contrast agent due to hydrolysis and, additionally, the transmetalation of the compound with iron present in the tissue matrix.

(O-13)

Development of HILIC-ICP-MS method for pharmaceutical complexes of Pt, Gd and I monitoring in drinking water resources

Joseph CÔME, Maïté BUENO, Florence PANNIER, Sandra MOUNICOU

Universite de Pau et des Pays de l’Adour, E2S UPPA, CNRS, IPREM, Institut des Sciences Analytiques et de Physico-chimie pour l’Environnement et les Matériaux, UMR5254, Hélioparc, 64053 Pau, France

The presence of contaminants in drinking water resources is a topic of great interest, yet certain types of compounds are still under-documented as for pharmaceutical residues for which there is a lack of knowledge about their occurrence in wastewaters and drinking water resources. These contaminants include organic complexes such as cancerostatic platinum compounds (CPC), iodine contrast media (ICM) and gadolinium-based contrasting agents (GBCA).
Because of their increasing use in recent years, their concentration in effluents and surface waters can now reach µgL^{-1} for iodine compounds and ngL^{-1} for platinum- or gadolinium-based compounds.
It is therefore necessary to develop precise analysis methods to identify and quantify the different chemical forms (speciation) of elements of interest (I, Gd, Pt). This step will ultimately make it possible to assess the impact of these compounds in natural waters.

The aim of this work is to develop an analytical method for the simultaneous determination of iodine-, platinum- and gadolinium-based complexes speciation at ultratrace levels (~ngL^{-1}). Our developments focused on the potential of hydrophilic interaction chromatography (HILIC), coupled with ICP-MS to separate and quantify 10 drug complexes, including 3 iodine-, 3 platinum- and 4 gadolinium- complexes. Three HILIC stationary phases (two zwitterionic with phosphorylcholine and ammonium-sulfonic acid stationary phases and one with a charged stationary phase based on silica) were compared with different mobile phase compositions (organic solvent percentage, salt concentration and pH) to evaluate their potential for metallodrug’s retention and separation.

HILIC phosphorylcholine zwitterionic stationary phase combined with a gradient mobile phase from 75:25 to 50:50 (A:B) ((A) 100% acetonitrile ; (B) 10 mmol.L^{-1} aqueous ammonium formate solution) was found optimal for the separation of the 10 targeted complexes with quantification levels between a few dozen (for GBCA and CPC) and a hundred (for ICM) of ng.L^{-1}.

The developed method was applied to environmental samples including surface waters, influents and effluents from urban wastewater treatment plants and, hospital effluent. Except in surface waters, our method allowed to detect and identify 2 ICM (iohexol and iopamidol from ~5 to ~20 µgL^{-1}) and 2 GBCA (gadoteric acid and gadobutrol from ~200 to ~800 ngL^{-1} and from ~30 to ~200 ngL^{-1} respectively). No CPC were detected.

(O-14)

The importance of non-target screening and mass balances for PFAS analysis: a job for elemental analysis

Jörg Feldmann, Viktoria Müller, Eleonora Matic, Raquel Gonzalez de Vega, David Clases and Andrea Raab

Analytical Chemistry, University of Graz, Universitätsplatz 1, 8010 Graz, Austria

The exposome research uses often non-targeted analysis or not-targeted screening in order to determine more than 10,000 compounds in water samples using RP-HPLC-ESI-HRMS. However, it is not possible to say which compounds are missed since they do not ionize or stick on the column. Another problem is the data mining and the determination of the level of confidence for their identification and also the quantification, especially if no standard is available. Here element analysis can help[1].

Mass balance approaches of an element is always used in speciation analysis to determine extraction efficiency and column recovery[2]. In terms of identification and quantification the online coupling of LC with HRMS and simultaneously to ICPMS will be demonstrated with current case studies with an emphasis on PFAS (per and polyfluorinated alkylated substances)3. Furthermore, the use of LA-ICPMS for fluorine mapping[4] and spICPMS/MS for fluorinated nano and microparticles.

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(O-15)

Seeing beyond LC-MS/MS in fluorine analysis for ski waxes

Viktoria Müller[1,2], Raquel Gonzalez de Vega[2], Erwin Rosenberg[3], Klaus Zangger[4],
David Clases[2], Andrew Kindness[1] and Jörg Feldmann[2]

[1] The James Hutton Institute, Craigiebuckler, Aberdeen AB15 8QH, Scotland, UK
[2] Analytical Chemistry, Institute of Chemistry, University of Graz, Universitätsplatz 1, 8010 Graz, Austria
[3] Institute of Chemical Technologies and Analytics, Vienna University of Technology, 1060 Vienna, Austria
[4] Organic Chemistry, Institute of Chemistry, University of Graz, 8010 Graz, Heinrichstraße 28/II

E-mail: Viktoria.Mueller@hutton.ac.uk

Per – and polyfluoroalkyl substances (PFAS) are of high concern due to their persistence in the environment. Routine PFAS analysis uses liquid chromatography - electrospray ionisation - tandem mass spectrometry (LC-ESI-MS/MS), which is limited to only those compounds that are easily ionisable with the electrospray. Moreover, LC-ESI-MS/MS is prone to interferences and matrix effects, which complicates the analysis.

Skiing, as one of the main sports in Northern Europe can contribute to the spread of PFAS into the environment, due to the use of fluorinated ski waxes. But how much PFAS do these fluorinated waxes contain? Another problem with the analysis is that fluorinated ski waxes consist mostly of semi-fluorinated alkanes, which are not measurable with LC-ESI-MS/MS, so how we can measure the contamination coming from the ski waxes?

ICP-MS/MS analysis can be carried out to indirectly measure fluoride (F^-) as barium fluoride $[BaF]^+$. The advantage of this technique is that it is fluorine specific and can ionize all types of PFAS in contrast to ESI. Thus ICP-MS/MS can be used to analyse a wider range of fluorinated compounds. However, the $[BaF]^+$ method is not the only fluorine specific method available. Utilising a helium plasma, for example as part of the gas chromatography atomic emission chromatography (GC-AED), fluorine can be analysed directly.

In the following study we analysed ski waxes to see the different PFAS distribution in them using a mass balance approach to see how much each technique contributes to the total fluorine content. We compared different techniques available for fluorine analyses, such as combustion ion chromatography, high resolution graphite furnace molecular absorption spectroscopy and fluorine nuclear magnetic resonance, to ICP-MS/MS to evaluate how ICP-MS/MS perform in comparison. Additionally, we also analysed two different extracts wax extracts with GC-AED to evaluate how much fluorinated alkanes and other fluorinated volatile compounds are present in the extracts.

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(O-16)

From rare trace elements to plastics – Application of ICP-MS and complementary techniques to study environmental impacts of the Anthropocene

Daniel Pröfrock [1], Anna Ebeling[1,2], Lars Hildebrandt[1,2], Tristan Zimmermann[1], Dominik Wippermann[1,2], Alexa Zonderman[1,2] and Ole Klein[1,2]

[1] *Helmholtz-Zentrum Geesthacht, Abteilung Anorganische Umweltchemie, Max-Planck Str. 1, Geesthacht, 21502, Deutschland*
[2] *Universität Hamburg, Department of Chemistry, Inorganic and Applied Chemistry, Martin-Luther-King-Platz 6, Hamburg, 20146, Deutschland*

E-mail: daniel.proefrock@hereon.de

Despite the ongoing reduction of emissions of contaminants into the environment, the coastal zones of the North Sea still belong to the most impacted ecosystems worldwide. In particular, the ongoing evolution of coastal zones into industrialized areas, e.g. due to extensive shipping or the construction of offshore wind farms within the framework of the ongoing energy transition in Europe even boosted the release of either known, but also of various new contaminants into the marine environment. This includes e.g. rare element groups such as the so called TCEs beside other threats such as micro and nano plastics (MP/NP), which gained a strong scientific and public interest during the last years.
The accurate analysis of such contaminants is in particular, of significance for public health concerns beside the overall future sustainable development and management of the coastal zones as required by EU wide legislation.

This contribution will focus on the role of new powerful analytical tools to study different unwanted chemical side effects on the marine environment due to the strong development in offshore wind energy production within the context of the ongoing European energy transition.

In particular, the application of ICP-MS/MS for interference handling as well as the role of new hyphenation approaches for routine ultra trace analysis of emerging contaminants at ng/L levels, which allows a systematically evaluation of the emission load caused by anthropogenic activities within the coastal zone, will be highlighted. In addition, complementary tool to target new pollutant groups such as plastics and paint particles will be discussed.

(O-17)

Evaluating the impact of soil composition on the mobility of tellurium species using ICP-MS

Katarzyna Kińska, Monika Sadowska and Beata Krasnodębska-Ostręga

Faculty of Chemistry, University of Warsaw, Pasteura 1, 02-093, Warsaw, Poland

E-mail: kkinska@chem.uw.edu.pl

Tellurium is a technology-critical element (TCE), with relatively limited data on its behaviour in the environment, especially the pedosphere. As with other TCEs, its more widespread use, especially in the production of new energy sources, will inevitably make tellurium a next-generation xenobiotic. The knowledge of Te/soil interaction, indicating physicochemical transformations in the soil environment and determining the mobility of different tellurium species is fundamental in the context of soil remediation processes and counteracting the mobility of Te in the soil environment from e.g. solar farms.

During the presentation, new information about time-dependent sorption/desorption of Te(IV) and Te(VI) in the presence of additives mimicking different soil systems will be presented. Model studies on tellurium spiked soils, differing in the content of Mn/Fe (hydr-)oxides and organic matter, were the source of important data about retention of Te species in soil. The fractionation study was based on single extractions of: easily leachable/exchangeable and carbonate fraction (0.1 mol L^{-1} CH_3COOH), reducible fraction (0.1 mol L^{-1} ascorbic acid in oxalate buffer), and organic fraction (30% H_2O_2 at 85°C followed by 0.5 mol L^{-1} CH_3COONH_4). Throughout the study, the elemental composition of soils and the presence of Te in the extracts were monitored by ICP-MS.

The obtained results revealed that Te(IV) and Te(VI) interact differently with the studied matrices, and that sorption efficiency is heavily dependent on soil composition, as well as the presence of compounds produced due to the rhizosphere activity (e.g. citrates). Organic matter plays some role in retention of Te, but with addition of iron oxides it was more efficient. The fractionation study also indicated significant differences in the mobility of both tellurium forms, pointing out the need to include speciation analysis in Te monitoring.

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(O-18)

Mechanistic understanding and biomedical implications of d-electron metal-protein interactions in dairy proteins

Tetiana Dyrda-Terniuk[1], Oleksandra Pryshchepa[1], Kinga Robotnik[2,1] and Paweł Pomastowski[1,2]

[1]Centre for Modern Interdisciplinary Technologies Nicolaus Copernicus University in Torun, Wileńska 4, 87-100 Toru, Poland
2Department of Inorganic and Coordination Chemistry, Faculty of Chemistry, Nicolaus Copernicus University in Torun, Gagarina 7, 87-100, Torun, Poland

This investigation delves into the complexation behaviors of d-electron metals with key dairy proteins—β-lactoglobulin, lactalbumin, lactoferrin, and specific casein fractions (αS1-, β-, κ-casein). Employing Inductively Coupled Plasma Mass Spectrometry (ICP-MS), we quantified these metals' binding affinities to the proteins, aiming to unveil their potential in biomedical contexts. Our approach integrated kinetic and isothermal studies, molecular docking, and quantum mechanical calculations to elucidate the mechanisms of metal-protein complex formation.

Initial protein characterization was conducted through MALDI-TOF MS and zeta potential measurements, supplemented by proteomic analysis using both classical digestion and microfluidic immobilized enzyme reactor (μ-IMER) techniques. This groundwork enabled a comprehensive examination of the kinetics and thermodynamics governing complex formation, applying models such as Henry, Freundlich, and Langmuir for interpretive depth. An array of spectrometric, spectroscopic, microscopic, and diffraction methods provided a detailed physicochemical profile of the resulting nanocomplexes.

These analyses revealed the surface functional groups, morphology, and stability of the proteins, with a particular focus on the influence of metal concentration. The alignment of experimental outcomes with theoretical predictions underscored distinct patterns of interaction; notably, a metallo-complex system was observed with zinc ions, while silver ions and β-lactoglobulin formed a hybrid system characterized by the generation of silver nanoparticles. The antimicrobial prowess of these nanocomplexes against various pathogens suggests a novel strategy for tackling antimicrobial resistance. Further, *in vivo* experiments on mice showcased the complexes' ability to promote wound healing, evidenced by improved tissue regeneration and weight gain within a ten-day period.

Through the lens of advanced spectrochemical analysis, our study sheds light on the sophisticated dynamics of d-electron metal interactions with dairy proteins. These findings pave the way for the development of innovative antiseptic formulations and offer promising avenues for enhancing wound healing processes, underlining the significant biomedical potential of metal-protein complexes.

This research was funded under the project title: *"Development of a preparative method for the isolation of biologically active lactoferrin,"* supported by the National Centre for Research and Development (NCBiR), under the LIDER program, grant number DPWP/LIDER-XIII/6/2023. Oleksandra Pryshchepa and Paweł Pomastowski are members of Torun Center of Excellence 'Towards Personalized Medicine' operating under Excellence Initiative-Research University. Tetiana Dyrda-Terniuk is a member of Emerging Fields "Cells as Experimental platforms and bioFACTories (CExFact)"

(O-19)

LIBS imaging: Recent advances and perspectives

Vincent Motto-Ros

Institut Lumière Matière, iLM UMR5306 Université Lyon 1-CNRS, 69622 Villeurbanne, France.

E-mail: vincent.motto-ros@univ-lyon1.fr

The imaging capability of laser-induced breakdown spectroscopy (LIBS) has a high potential in various domains including biology, industry, geology and medicine (c.f. figure 1) [1,2]. This approach can be distinguished by its ease in use, multi-elemental capability, detection of light elements, as well as operation at ambient conditions. This is furthermore the only all-optical technique providing space-resolved elemental information with ppm-scale sensitivity and µm-range resolution. These advantages, make LIBS imaging very attractive to be used in research laboratories for routine investigations.

However, advanced technological solutions must be found for this application since elemental imaging requires high sensitivity, sharp spatial resolution, high speed of acquisition as well as the ability to process a huge quantity of data. In this presentation, we will summarize the recent progresses made in the Light and Matter Institute concerning the implementation of the LIBS imaging. In particular, different examples of breakthrough applications, such as biomedical (figure 1.a and b) or industrial (figure 1.c and d) will be shown with the aim of illustrating the specificities and the great potential of LIBS imaging. Different perspectives will be finally discussed.

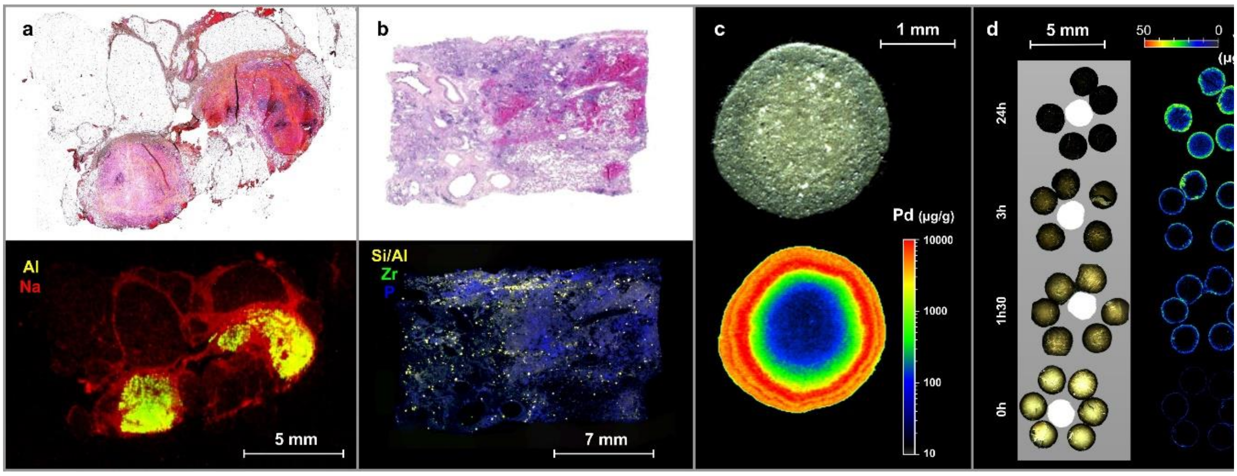

Figure 1. (a), Histologic analysis combined with elemental imaging that identified the metal responsible for a foreign body granuloma in a young patient at CHUGA. (b), Histological and multi-elemental images of a lung biopsy from a patient who worked in the World Trade Center rubble removal. (c) Demonstration of the distribution of the active element Pd of a catalyst supported on alumina, image representing a dynamic of 3 orders of magnitude in concentration. (d) Kinetics of impregnation of a heavy metal, vanadium, during petrochemical treatments.

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(O-20)

Multimodal chemical imaging for biomedical applications

Uwe Karst, Katharina Kronenberg, Christine Verlemann, Peter Niehaus and Michael Sperling

Institute of Inorganic and Analytical Chemistry, University of Münster, Corrensstr. 48, 48149 Münster, Germany

E-mail: uk@uni-muenster.de

The analysis of chemical elements and their species are highly important to study their properties, including the distribution in human, animal or plant tissue samples. Applications include (metallo)pharmaceutical chemistry, (nano)toxicology and biomaterials research.

In this presentation, differently graded liver tumor tissue of animals after exposure to a liver-specific gadolinium-based contrast agent and Cisplatin as antitumor agent was investigated to study the chemical changes associated with the expansion of the tumor and the metallopharmaceuticals.

Optical microscopy is first used to identify tissue areas, including tumor tissue, lesions and seemingly unaffected tissue. An overview of the distribution of highly abundant elements was obtained by micro-X-ray fluorescence analysis (µXRF). Laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) provides quantitative distribution information on gadolinium, platinum and several endogenous elements including iron, copper and zinc based on calibration using gelatin-based external standards.

Complementary matrix-assisted laser desorption/ionization-mass spectrometry (MALDI-MS) with highly resolving mass analyzers and integrated ion mobility spectrometry (IMS)-based separation delivers distribution information on lipids in different tissue areas. Infrared (IR) microscopy, on the other hand, is a highly useful tool to distinguish between different tissue types. For example, high glycogen signals in lesions reveal expansion strategies of the tumor. Chemometrics approaches as Uniform Manifold Approximation and Projection for Dimension Reduction (UMAP) prove to be highly useful tools for precise tissue assignment based on chemical information.

(O-21)

High-resolution bioimaging with nanoscale secondary ion mass spectrometry and complementary techniques

Dirk Schaumlöffel, Iris H. Valido and Maria Angels Subirana

CNRS/Université de Pau et des Pays de l'Adour, E2S UPPA, IPREM UMR 5254, Hélioparc,
2, avenue du président Angot, F-64053 Pau, France

E-mail: dirk.schaumloeffel@univ-pau.fr

New instrumental and methodological analytical developments for bioimaging lead to important progress for the investigation of biochemical functions, biosorption and bioaccumulation processes. Nanoscale secondary ion mass spectrometry (NanoSIMS) relies on the sputtering of ions from a solid surface by focused positive or negative primary ion beams and the subsequent analysis of the produced secondary ions by a mass spectrometer under high vacuum. Thanks to its high spatial resolution down to 50 nm, NanoSIMS is perfectly suited to localize the distribution of chemical elements with high spatial resolution in biological tissues and at cellular and subcellular level. In addition, a RF plasma oxygen primary ion source offers high sensitivity and high spatial resolution for trace metal detection. Furthermore, we develop correlative bioimaging where NanoSIMS enables a nanoscale zoom of the same areas previously imaged by LA-ICPMS at microscale. Synchrotron-based X-ray techniques complete the analytical approach for imaging and in-situ speciation.

This lecture covers analytical developments and applications of NanoSIMS and related techniques for bioimaging and speciation. In particular, NanoSIMS for localization of metals and nanoparticles in cells and tissues will be presented. This will be demonstrated by recent examples from our research such as distribution and dissolution of barium sulfate nanoparticles in human lungs cells, the localization and speciation of zinc in zinc-accumulating yeast, and the correlative bioimaging of essential trace metals in the context of trace metal supplementation in animal nutrition. Finally, challenges with this correlative bioimaging approach, will be addressed, and first strategies for quantitative SIMS imaging will be discussed.

(O-22)

Enhanced stable isotope tracking of elemental fluxes: From biological metabolism to industrial processes

Johanna Irrgeher [1], Alexander Epov[1], Susanne Michelic[2,3], Markus Puschenreiter[4], Kathrin Thiele[2,3], Stefan Wagner[1], Andreas Zitek[5,6] and Thomas Prohaska[1]

[1] Montanuniversität Leoben, Chair of General and Analytical Chemistry, 8700 Leoben, Austria
[2] Montanuniversität Leoben, Chair of Ferrous Metallurgy, 8700 Leoben, Austria
[3] Christian Doppler Laboratory for Inclusion Metallurgy in Advanced Steelmaking, Montanuniversität Leoben, 8700 Leoben, Austria
[4] University of BOKU Vienna, Institute of Soil Research, 3430 Tulln, Austria
[5] University of Natural Resources and Life Sciences Vienna, Department of Chemistry, 1190 Vienna, Austria
[6] FFoQSI GmbH, Technopark 1D, 3430 Tulln an der Donau, Austria

E-mail: johanna.irrgeher@unileoben.ac.at

Isotopically enriched spikes of natural occurring elements have experienced increasing interest as monitors of elemental uptake and distribution as well as unique markers of products or goods. For example, isotopic spikes have been administered to biological systems for the monitoring of ecosystem fluxes or as promising tool to investigate elemental behavior and deposition during metabolism in plants, animals or humans. However, the potential of using specific isotopic spikes as unique markers has not been fully exploited yet. In this context, (multiple) isotopic spikes offer the opportunity to provide unique fingermarks for valuable goods, artefacts, hazardous substances (such as explosives) or to study and better understand industrial processes by marking specific steps in the production process. Isotope pattern deconvolution (IPD) can be applied as mathematical tool based on multiple linear regressions allowing for deconvolving these isotope pattern without knowing the quantities of different isotope sources incorporated and mixed into the sample as well as the degree of impurities and species-interconversion.

In this presentation, we will explore selected studies where Mg, Ni, Sr, and Pb isotope spikes have been utilized across a spectrum of applications, ranging from biological, clinical, and medical fields to material sciences and sophisticated industrial operations, highlighting the expansive potential and versatility of enriched isotopic spikes:
In our laboratory, we started to investigate the utility of this method by applying an ^{84}Sr single and an ^{86}Sr/^{84}Sr double spike for transgenerational fish marking. We traced the central otolith regions of female freshwater fish progeny treated with varying concentrations of ^{84}Sr and ^{86}Sr, enabling the potential assignment of the offspring to their individual mothers. The capability of enriched ^{204}Pb was evaluated for monitoring trace levels of Pb in humans within a clinical study assessing the impact of purified clinoptilolite tuff on enteral lead uptake in adults. The method facilitated the deconvolution of isotope patterns from measured isotope ratios, without the need to know the quantities of various isotope sources incorporated and mixed into the sample at levels below 1 pg ^{204}Pb/g blood. In a pilot study within the field of pediatric surgery, metal Mg enriched with ^{26}Mg was implanted into rats to examine bone implant degradation and Mg mobilization *in vivo*. Extending our scope, we are currently using ^{26}Mg to trace the sources of non-metallic inclusions in steel. In a current study in the field of plant physiology, ^{61}Ni isotopes are applied to investigate the Ni uptake in hyperaccumulating plants under greenhouse conditions.
The highlights and capabilities of the method will be presented along with its challenges.

(O-23)

Recent advances in isotopic analysis using MC-ICP-MS: Analytical method developments and applications

Lana Abou-Zeid[a], Laura Suárez-Criado[a,b], Maria Alessia Vecchio[a,c], Kasper Hobina, Marta Costas Rodríguez[d], Eduardo Bolea-Fernandez [a,e], Marco Grotti[c] and Frank Vanhaecke[a]

[a] *Ghent University, Department of Chemistry, Atomic & Mass Spectrometry – A&MS research group, Ghent, Belgium*
[b] *University of Oviedo, Department of Physical and Analytical Chemistry, Julian Claveria 8, 33006-Oviedo, Spain*
[c] *University of Genoa, Department of Chemistry and Industrial Chemistry, Genoa, Italy*
[d] *Centro de Investigación Mariña, Universidade de Vigo, Departamento de Química Analítica y Alimentaria, Grupo QA2, 36310 Vigo, Spain*
[e] *University of Zaragoza, Aragon Institute of Engineering Research (I3A), Department of Analytical Chemistry,Zaragoza, Spain*

Several decades ago, isotopic analysis emerged as a powerful tool to identify natural isotopic variations for several elements in the context of in-depth investigation of natural processes, mainly in the fields of geology & cosmology, environmental sciences, but also in biology and medicine. In most cases, multicollector inductively coupled plasma-mass spectrometry (MC-ICP-MS) is the technique of choice for conducting such analysis as it offers high measurement precision (down to 0.001% RSD) due to the simultaneous detection of all isotopes of the targeted element, enabling very small changes in isotopic compositions to be revealed and quantified.

Despite the advantage that MC-ICP-MS offers in terms of precision, it suffers from several drawbacks related to its limited sensitivity, especially when the amount of analyte available for measurement is a limiting factor. Decreasing the analyte concentration measured can be an option to address this limitation, however it is often accompanied with a degradation of measurement precision due to the low signal intensity. Recent improvements in the instrumentation's capabilities and the development of novel analytical approaches allowed these limitations to be successfully tackled. Alternative introduction systems such as desolvating units or hydride/vapor generation systems enhance the analyte introduction efficiency and therefore, the signal intensity. In addition, Faraday cup amplifiers equipped with 1013 ohm resistors were recently introduced in MC-ICP-MS instrumentation. The latter offer a notably enhanced signal-to-noise ratio for ion beams at low signal intensities compared to the standard 1011 Ω resistors, which enabled high-precision isotopic analysis to be conducted at low analyte concentration levels with sufficient precision [1,2].

In addition to limitations related to the sensitivity, MC-ICP-MS measurement of some elements can be challenging due to spectral overlap. Increased resolution of the commercially available MC-ICP-MS instrumentation allowed, in some cases, resolving these interferences[3], while in other cases the resolution needed is still lacking. In these specific cases, reducing spectral interference can be achieved through introducing specific gases into the plasma for suppressing the signal of the interfering ions (mainly argon-based)[4] or through simply revisiting fundamental aspects of the plasma for sampling in regions with minimal formation of the interfering ion type.

Recent analytical method developments carried out in the A&MS group in the field of isotopic analysis, in which we addressed all the above-mentioned limitations, will be presented and discussed, as well as further potential improvements in order to ascertain accurate, precise and ultrasensitive MC-ICP-MS measurements.

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(O-24)

Drug counterfeits – identification via element isotope ratios and trace elemental patterns

Stefan Stürup[a] and Else Holmfred[b]

[a]Department of Pharmacy, University of Copenhagen, Denmark
[b]Green Earth Sciences, Stanford University, California, USA

E-mail: stefan.sturup@sund.ku.dk

More than 50% of the European population take medicine on a regular basis. Many of these drugs contain elemental impurities originating from eg. residual catalyst or impurities. Since elemental impurities do not have any therapeutic effect, their level in drug products should be below acceptable limits as described in the ICH guideline on elemental impurities. For this purpose, ICPMS based quantitative methods are now well established and applied on a routine basis. An increasing problem worldwide is drug counterfeits, and the World Health Organization estimates that up to 10% of drugs in low- and mid-income countries are counterfeits yearly causing thousands of deaths. Identification of drug counterfeits is often challenging and there is a need for novel analytical methods and data evaluation strategies. The purpose of this project is to combine elemental (20+ elements) and isotope ratio (Sr, C, H) determinations to characterize genuine drugs eg. variations in formulation, production methods, and batches, and at the same time ensure that the drugs are in compliance with the ICH guideline. In addition, combined elemental and isotope ratio patterns will applied in an attempt to identify drug counterfeits through multivariate data analysis. It is expected that drug counterfeits will exhibit different patterns, as they are often produced in a more simple and less controlled laboratory environment. During this presentation preliminary results from the project will be presented.

Although drug tablets is a well characterized formulation with a known API (active pharmaceutical ingredients) and a known number of excipients it is often difficult to fully digest tablet powder with acids (HNO_3, HCl etc) and obtain a clear solution without precipitates prior to ICPMS analysis as most tablets contain excipients as titanium oxide, silicium oxide, iron oxide, and Mg-stearate. Results on the digestion of a range of different ibuprofen tablets with different microware digestion methods are presented and their effectivity evaluated via residual cardon is discussed.

As a model drug, ibuprofen is used for method establishment and method validation before applying the new techniques to other pharmaceuticals. The elemental impurity profiles obtained for the Ibuprofen tablets (ICPMS) are combined with $^{13}C/^{12}C$ isotope ratios (IR-MS) in order to differentiate the Ibuprofen tablets based on formulation and geographical origin. The differences seen originate primarily from the use of different excipients as they come with specific elemental impurities and carbon isotope patterns, which can be applied for characterization of specific formulation and production methods.

Finally, preliminary results from the use of the $^{87}Sr/^{86}Sr$ isotope ratio as a formulation/production marker for carbonate based antacids are presented. The $^{87}Sr/^{86}Sr$ isotope ratio is determined as $^{87}SrO/^{86}SrO$ using ICP-MSMS with a precision better than 0,1 %RSD.

(O-25)

Development and comparison of analytical methods for the simultaneous measurement of Sr and Pb isotope ratios

F. Bruschi[a], A. Lores Padin[b], E. Bolea-Fernandez[c], K. Hobin[b], D. Cappelletti[a] and F. Vanhaecke[b]

[a] *Department of Chemistry, Biology and Biotechnology, University of Perugia, Via Elce di Sotto 8, 06123, Perugia, Italy*
[b] *Department of Chemistry, Atomic & Mass Spectrometry - A&MS Research Group, Ghent University, Campus Sterre, Krijgslaan 281-S12, BE-9000, Ghent, Belgium*
[c] *Department of Analytical Chemistry, Aragon Institute of Engineering Research (I3A), University of Zaragoza, 50009, Zaragoza, Spain*

Stable isotope ratios of chemical elements contain valuable information concerning the natural and/or anthropogenic processes that have affected an environmental sample over time, complementing the information given by multi-elemental analysis. For this reason, isotopic analysis has become an indispensable tool for a wide range of applications.

The ICP-MS techniques have been proven to be very versatile; they allow for quantitative determination of elements at (ultra)trace levels, even those that are difficult to ionize, after a simple sample preparation procedure, while guaranteeing a wide linear dynamic range, good stability, and a short analysis time. Because of the high precision required when performing isotopic analyses, multi-collector (MC) ICP-MS stands as the established method due to its simultaneous acquisition capability. In contrast, quadrupole-based ICP-MS operates sequentially, while TOF operates in a quasi-simultaneous mode, therefore, these techniques are not commonly used for isotopic analysis.

In this study, we assess the performance of tandem ICP-MS and ToF ICP-MS for the simultaneous analysis of Sr and Pb isotope ratios. With this aim, a set of geological certified reference materials (CRMs) was microwave-digested and analyzed with the two instruments and with an MC-ICP-MS unit (benchmark) as a reference method. ToF ICP-MS requires a chromatographic isolation of the target elements before the measurement, just as the MC-ICP-MS. This time-consuming step can be avoided using tandem ICP-MS, thanks to its chemical resolution, which allows the removal of interfering species. Specifically, it was possible to optimize a method granting good precision and accuracy using the reactive gas CH3F. In the case of ToF ICP-MS, the quasi-simultaneous acquisition of all the analytes was possible (each 46 us the whole mass spectrum is recorded) allowing a much faster isotopic analysis measurement. Additionally, two different mass-bias correction methods were proposed and applied to the results obtained with both instruments. In the first method, the sample-standard bracketing was performed, using a NIST concentration that was in the middle of the sample concentration range. The second method consisted of the analysis of three NIST standard solutions with increasing concentrations before and after a set of three samples; each sample was then bracketed with the standard having a similar concentration. The results obtained using the two instrumental setups as well as the two bracketing methods were comparable.

(O-26)

From theory to practice: Implementing cutting-edge ICP-MS for swift radiochemical analysis

Jixin Qiao

Department of Environmental and Resource Engineering, Technical University of Denmark (DTU Sustain), DTU Risø Campus, 4000 Roskilde, Denmark

E-post: jiqi@dtu.dk

Efficient and precise analysis of radionuclides is of great significance in various fields such as environmental and food radioactivity monitoring, radioecological risk assessment, and decommissioning of nuclear facilities and waste management. While most γ-emitting radionuclides can be directly measured after simple sample processing, the analysis of "difficult-to-measure" radionuclides, such as α, β, or low-concentration γ-emitting radionuclides, has always been a challenge in the field of radiochemical analysis. The Radioecology and Tracer Studies (RTS) research group at DTU Sustain in Denmark has been dedicated to developing and optimizing analytical methods for "difficult-to-measure" radionuclides in various types of samples for environmental/food monitoring and operation and decommissioning of nuclear facilities.

In recent years, the development of novel inductively coupled plasmas mass spectrometry (ICP-MS) techniques has contributed greatly to achieve rapid, efficient, and simultaneous multi-isotope measurement of difficult-to-measure radionuclides in environmental, biological, and radioactive waste samples. This paper aims to briefly introduce the research progress in recent years in ICP-MS method development for difficult-to-measure radionuclides in environmental monitoring and nuclear decommissioning in Denmark.

(O-27)

Tracing landfill leachate in environmental samples using a triple isotope approach - δ^{13}C, δ^{11}B and $^{87/86}$Sr in Norwegian landfill leachate

Christian Schöpke[1,2] Ingar Johansen[1], Vani Devegowda[1], and Julianne Hem[2]

[1]Institute for Energy Technology (IFE), 2007 Kjeller, Norway
[2]Norwegian University of Life Sciences (NMBU), 1432 Ås, Norway

Leachate from industrial and municipal solid waste landfills is a complex fluid characterized by high levels of organic and inorganic pollutants. As such, there are often requirements for characterization and treatment of leachate from landfills due to their potential to contaminate surface and groundwaters. However, many landfills have insufficient control over the water flowing in and out of the system, and pollution is a common occurrence. The aim of this contribution is to demonstrate the effectiveness of the isotope systems of boron (δ^{11}B), strontium ($^{87/86}$Sr), and dissolved inorganic carbon (δ^{13}C-DIC) as tracers of Norwegian landfill leachate.

The studied isotopic systems were selected based on parameters including types of waste and composition of leachate, local geology, local hydrology, and the presence and type of other nearby pollution sources. In this study, we present the results of 15 years of δ^{13}C-DIC measurements at selected Norwegian landfills based on annual sampling. Additionally, we present for the first time δ^{11}B and $^{87/86}$Sr measurements of landfill leachate from Norwegian landfills, in combination with those from groundwater and freshwater bodies near the landfill sites. The δ^{11}B and $^{87/86}$Sr measurements were performed on a multi-collector ICP-MS instrument after matrix removal using ion exchange chromatography, and we discuss challenges in δ^{11}B determination as well as evaluate the utility of δ^{11}B and $^{87/86}$Sr as leachate tracers at the studied sites as supplements to δ^{13}C-DIC. Increased understanding of the behavior of landfill leachate can lead to more targeted remediation measures and a benefit for landfill operators, local communities, and the environment.

(O-28)

Determining the precise atomic abundance of ^{107}Pd in a nuclear waste sample using the isotope dilution technique and multi-collector (MC)-ICP-MS

Majd SHMEIT, Marina FAURE, Celine GAUTIER and Hélène ISNARD

Université Paris-Saclay, CEA, Service de Physico-Chimie, 91191, Gif-Sur-Yvette, France

Palladium-107 is a long-lived radiogenic isotope with a half-life of 6.5 (±0.3) million years, and is produced by the fission of ^{235}U in nuclear reactors. Consequently, there is a need to precisely determine the atomic abundance ^{of} ^{107}Pd in nuclear waste prior to its long-term storage and in referral to international guidelines. Determining the atomic abundance of ^{107}Pd allows evaluating its contribution to radioactivity in a given material. In this study, we describe the methodology to precisely quantify the atomic abundance and concentration of ^{107}Pd in a solution of nuclear waste. The nuclear waste sample was provided in the form of a liquid solution purified beforehand by liquid-liquid extraction and ion chromatography. The quantification of ^{107}Pd is based on the isotope dilution technique by mixing a fraction of the nuclear waste with a tracer enriched in ^{108}Pd (metal powder; Eurisotop – France). This is followed by measuring all Pd isotope ratios (^{102}Pd/^{108}Pd, ^{104}Pd/^{108}Pd, ^{105}Pd/^{108}Pd, ^{106}Pd/^{108}Pd, 1^{07}Pd/^{108}Pd, ^{110}Pd/^{108}Pd) using a MC-ICP-MS (Neptune Plus, Thermo Scientific).

Initially, the enriched tracer was dissolved using a concentrated solution of HNO3-HCl. Then, we certified the isotopic composition and the atomic abundance of Pd isotopes in the enriched tracer by reverse isotope dilution using the natural Pd NIST 3138 standard. All working solutions (nuclear waste sample, enriched tracer, and NIST 3138) were prepared at roughly 28 ppm in 10% HCl, and then used to prepare the isotope dilution mixtures. Isotopic measurements were done in diluted solutions at 200 ppb in 2% HNO3. We present the preliminary results obtained on the enriched tracer and the nuclear waste sample.

The measured isotope ratios in the enriched tracer allowed to determine the atomic abundance of ^{108}Pd at 98.759 ±0.007%, the uncertainty being calculated at k=2 following the Kragten method [1]. In addition, the measured isotope ratios demonstrated a concentration of total Pd in the working solution of the tracer at 26.62 ±0.03 ppm (1.3 ‰, k=2). This result was obtained from measuring the Pd isotope ratios in the tracer as well as in 7 mixtures prepared from varying volumes of the tracer and NIST 3138. Moreover, the precision on isotope ratios measured in the nuclear waste sample was lower than 0.04% (n=10); the uncertainty being two-times the standard deviation of analytical measurements. In the perspectives, further isotopic analyses will be conducted on isotope dilution mixtures (nuclear waste sample and enriched tracer). The nuclear waste sample will be also re-purified by extraction chromatography in order to validate the absence of ^{107}Ag. The latter isotope is produced from the radioactive disintegration of ^{107}Pd, and can interfere on the mass 107 (isobar) during mass spectrometer analyses.

[1] KRAGTEN, J. Tutorial review. Calculating standard deviations and confidence intervals with a universally applicable spreadsheet technique. Analyst, 1994, vol. 119, no 10, p. 2161-2165.

(O-29)

Re-determination of the absolute $^{13}\text{C}/^{12}\text{C}$ isotope ratio for Vienna Peedee Belemnite (VPDB) isotope-delta scale

Dmitriy Malinovskiy,[1] Philip J.H. Dunn,[1] Nives Ogrinc,[2] Doris Potočnik,[2] Lukas Flierl,[3] Olaf Rienitz,[3] Dipayan Paul,[4] and Harro A. J. Meijer and [4] Heidi Goenaga-Infante[1]

[1] *National Measurement Laboratory, LGC Limited, Teddington, UK*
[2] *Department of Environmental Sciences, Jožef Stefan Institute, Ljubljana, Slovenia*
[3] *Physikalisch-Technische Bundesanstalt, Braunschweig, Germany*
[4] *Centre for Isotope Research (CIO), University of Groningen, Groningen, Netherlands*

Variations in the stable isotope composition of carbon are used in diverse fields, including environmental science, biology, food authentication and forensic applications. Results of $^{13}\text{C}/^{12}\text{C}$ ratio measurements are commonly reported as delta values, relative to a measurement standard selected as a zero point to anchor the international carbon isotope-delta scale. Although the delta notation is a convenient way to report small differences in isotope composition, a drawback associated with it is that traceability of measurement results is established to scale-defining artefacts, lacking a straightforward link to the International System of Units (SI). The knowledge of absolute isotope ratios is needed to allow traceability of isotope ratio data to the SI. That is why the accurate and precise determination of absolute $^{13}\text{C}/^{12}\text{C}$ isotope ratios, *i.e.*, $n(^{13}\text{C})/n(^{12}\text{C})$, of carbon isotope-delta scale artefacts is important for ensuring data compatibility between different laboratories and over long periods of time.

In this work, we describe a strategy towards re-determination of the $n(^{13}\text{C})/n(^{12}\text{C})$ isotope ratio of VPDB zero point with a reduced measurement uncertainty by building upon previous work. To achieve this, $n(^{13}\text{C})/n(^{12}\text{C})$ isotope ratios of D-glucose samples were measured by MC-ICP-MS calibrated by gravimetric mixtures of isotopologues and $\delta(^{13}\text{C})_{\text{VPDB}}$ values of the same samples by IRMS calibrated by reference materials traceable to VPDB. A linear regression constructed by using these data allowed us to estimate the $n(^{13}\text{C})/n(^{12}\text{C})$ isotope ratio at the point of VPDB scale where the carbon isotope delta is equal to zero. A ten-fold reduction in the uncertainty of the determined $n(^{13}\text{C})/n(^{12}\text{C})$ ratio of VPDB zero point over previous best measurement was achieved after implementation of an improved experimental design. The isotope ratio data obtained were verified by interlaboratory measurements, involving leading expert laboratories in the field.

References:
Malinovsky D, Dunn PJH, Holcombe G, Cowen S, Goenaga-Infante H. Development and characterisation of new glycine certified reference materials for SI-traceable $^{13}\text{C}/^{12}\text{C}$ isotope amount ratio measurements. *J. Anal. At. Spectrom*. 2019, 34(1), 147-159.

(O-30)

Direct determination of radium-226 and radium-228 in groundwater with the NexION 5000 multi-quadrupole ICP-MS

Michael Petrich[1] and Karl Andreas Jensen[2]

[1] *PerkinElmer GmbH*
[2] *Centre for Environmental Radioactivity (CERAD), Norwegian University of Life Sciences, Ås, Norway*

The WHO drinking water guidance level for ^{226}Ra is 1 Bq/L and for ^{228}Ra 0.1 Bq/L. The U.S. Environmental Protection Agency (USEPA) has set a combined MCL (maximum contaminant level) for $^{226+228}$Ra by 0.185 Bq/L. In the EU, the Council Directive 2013/51/EURATOM specifies activity concentrations of 0.5 Bq/L ^{226}Ra (14 pg/L) and 0.2 Bq/L ^{228}Ra (0.02 pg/L). The required detection limits (LOD) of ^{226}Ra in drinking water is 0.04 Bq/L (1 pg/L).

Measurement of ^{226}Ra in environmental samples can be done by alpha spectrometry, gamma spectrometry, liquid scintillation spectrometry (LSS) and mass spectrometry (ICP-MS, TIMS, AMS). Radiometric determination usually involves lengthy, work-intensive sample preparation. Counting time in these techniques is typically hours to days for one sample, which together with the potential need for prior concentration, radiochemical separation, and/or waiting for equilibration with daughter nuclides for up to 30 days notoriously impedes rapid processing of samples.

The ISO draft standard ISO/DIS 4685 describes an ICP-MS method for the determination of ^{226}Ra applicable in the event of an emergency. It recommends a first preconcentration of ^{226}Ra before undergoing purification from potential interferent by passing solution through a cation exchange resin followed by a crown-ether based extraction.

This work describes the direct measurement of ^{226}Ra in groundwater samples in less than 3 min without any preconcentration or matrix separation. Detection can be performed on mass using NH_3 at 226 amu or alternatively via a mass shift reaction with N_2O at 242 amu. Both cell gas reactions are explained using product ion and precursor ion scans. The calibration range extends from 1 - 500 ppq, MDL 0.6 ppq with both reaction gases. Groundwater samples with elevated Ra concentrations from the area of former East German uranium mining as well as groundwater from Norwegian deep wells were measured. The results of the ICP-MS/MS measurement are in excellent agreement with those of alpha spectrometry for all samples.

Despite comparable sensitivity and detection limits, this agreement can neither be achieved with NH_3 nor with N_2O in single quad mode for all real samples, even under optimized cell conditions. The relevant interferents are discussed for both cell gases.

^{228}Ra has a factor 276 shorter half-life than ^{226}Ra and therefore requires monitoring in the ppqt (fg/L) range. This is not possible without prior enrichment. Nevertheless, the method also provides initial information on ^{228}Ra within the rapid emergency response.

(O-31)

Environmental reconstruction studies through the elemental analysis of honeysamples with radiocarbon based age determination

Zsófi SAJTOS[a], Ágota Zsófia RAGYÁK[a], Tamás VARGA[b], Zita GAJDOS[a], A.J. Timothy JULL[b,c,d], Zsuzsa LISZTES-SZABÓ[b], Mihály MOLNÁR[b], Gábor BELLÉR[e] and Edina BARANYAI[a]

[a] Environmental Analytical Research Group, Department of Inorganic and Analytical Chemistry, Faculty of Science and Technology, University of Debrecen, Egyetem Square 1, H-4032 Debrecen, Hungary
[b] International Radiocarbon AMS Competence and Training (INTERACT) Center, Institute for Nuclear Research, Debrecen, H-4026, Bem Square 18/c, Hungary
[c] Department of Geosciences, University of Arizona, Tucson, AZ 85721 USA
[d]University of Arizona AMS Laboratory, Tucson AZ 85721 USA
[e] Department of Environmental Engineering, University of Debrecen, Faculty of Engineering, 2-4 Ótemető Street, H-4028, Hungary, Debrecen
E-mail: sajtos.zsofi@science.unideb.hu

Honeys are excellent indicators of the environment, several studies show that the elemental content of honey entirely depends on the botanical and geographical origin, but the information is incomplete regarding time-dependent composition changes. Sixty-five honey samples with different botanical origin (acacia, sunflower, canola and forest honeys) were collected between 1958-2018 and analyzed for elemental composition by microwave plasma optical emission spectrometry (MP-AES). The elemental analysis was coupled with independent dating method by accelerator mass spectrometry (AMS) to determine the real age of the honey samples and test the possibility of radiocarbon based dating of bee products, which has not been applied before.

According to the analytical measurements and statistical analysis, we concluded that bee products regardless the type provide useful environmental information of the previous decades, such as the decreasing trend of airborne Pb emission can be traced. We have proven that honey preserves information of previous times and thus can be applied as an environmental indicator in reconstruction studies by analyzing the non-degradable mineral content.

Good agreement was observed between the radiocarbon content of the acacia honeys and the atmospheric bomb-peak that was used for calibration. However, radiocarbon results agree less with the atmospheric bomb peak, random offsets were observed in the specific radiocarbon activity of the honey samples with agricultural field origin (canola and sunflower honeys). Fourier Transformation Infrared Spectrometry (FTIR-ATR) was also used to gain a deeper knowledge regarding the organic material composition of the honey samples. Results indicate that neither the inorganic composition nor the organic constituents show corresponding pattern to the AMS deviation. Even the 5-(Hydroxymethyl)furfural content following the expected age trend in acacia honeys has no such pattern in present sample series. This work indicates that canola and sunflower honey samples are not as reliable materials for radiocarbon dating as acacia honeys. Thus, the complex application of honey samples for environmental reconstruction requires the species-separated investigation of bee products to reveal their adaptability for assessment approaches.

Acknowledgements
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(O-32)

What to expect from modern high-temperature microwave acid digestion

Markus Michaelis and Christian Trampitsch

Anton Paar GmbH, Graz, Austria

Atomic spectroscopy has seen significant advancements, transitioning from atomic absorption to ICP-MS. However, sample preparation for trace element analysis often remains a tedious, multi-step process, sometimes even in open vessels.

This presentation explores high-temperature microwave acid digestion, a faster and more efficient alternative. We will delve into the fundamentals, including reaction temperatures, reagent mixtures, and their impact on vessel pressure.

We will discuss how to assess digestion quality for diverse sample types, ranging from organic materials to minerals. Advancements in instrumentation and vessel design will be highlighted, showcasing how users benefit from accelerated sample preparation workflows.

Finally, get a glimpse of real-life examples, including high-performance digestion of graphite and high-throughput analysis of various food samples in a single run. We will also explore advanced features offered by modern acid digestion instruments.

This presentation emphasizes how high-temperature microwave acid digestion empowers researchers with a faster, more streamlined approach to sample preparation, ultimately leading to improved analytical efficiency.

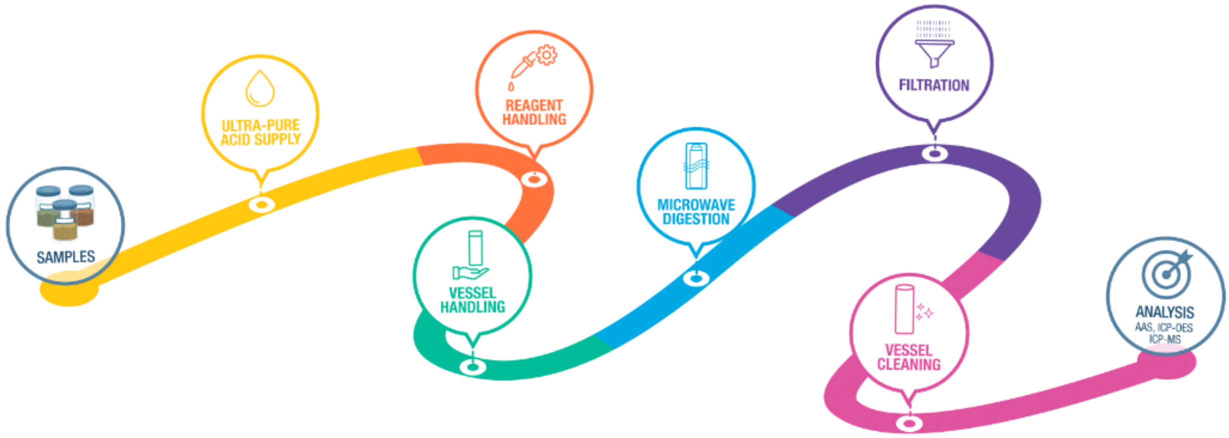
(O-33)

Overview of a “total workflow” approach to sample prep and why it matters

Laila Kvalheim[1] and Gianpaolo Rota[2]

[1] *HolgerHartmann, Lanhus, Norway*
[2] *Milestone, Sorisole, Italy*

The 'total workflow' approach to sample preparation for elemental analysis examines not only the evolution of the microwave digestion process, but also other workflow steps that are critical to meeting laboratory performance requirements. By optimizing these steps, laboratories are better prepared to overcome the challenges they face and avoid workflow disruptions that impede the analysis process and can have additional effects.



(O-34)

Analysis of line rich matrices – Challenges and possible solutions

Petar Ivanov, Olaf Schulz and Marcus Gantenberg

Spectro Analytical Instruments GmbH, Boschstraße 10, 47533 Kleve, Germany
E-mail: petar.ivanov@ametek.com

ICP-OES instruments are widely used for the analysis of different type of samples. Their multielement capability, combined with very good sensitivity and matrix tolerance make them the perfect choice in the modern labs.

Line rich matrices, however, can be challenging due to possible interferences. Metals like Cu, Co, Ni, Fe etc. are producing a high number of spectral interferences.

Viewing the plasma radially provides generally lower sensitivity, but matrix effects are greatly reduced. Therefore, the radial observation technique is typically chosen in the case of samples with high TSD (total dissolved solids) concentrations, for the routine analysis of organic solutions or where high stability and precision is required.

The possibility to view the plasma both axial and radial at the same instrument (dual view) seems to solve all disadvantages, but those techniques are a compromise since only one light path is direct.

The MultiView technology combines the benefits of the axial and radial view in one instrument.

Using a Dual Side-on interface is a new approach which provides the possibility of improvement of the radial plasma.

In this study the different plasma views will be critically compared for the analysis of more complex matrices, like volatile organics, high salt samples, Li-Ion battery materials etc. Sensitivity, linearity, robustness and stability will be reported.

The use of the different views with hyphenated techniques will be discussed as well.

The different views of the plasma and the torch orientation play an important role for the analytical capabilities of the instrument and the performance in difficult matrices and complex samples.

(O-35)

Automated high throughput analysis of large batches of mining discovery samples by LA-ICP-MS

Daniel Fliegel[3], Ruth Merrifield[1] and Rob Hutchinson[2]

[1] PerkinElmer Inc., Woodbridge, ON, Canada
[2] Elemental Scientific Europe Ltd., Huntingdon, UK
[3] PerkinElmer Norway AS, Oslo, Norway

The importance of identifying economically viable deposits of rare earth elements (REE) has grown significantly. REE are integral parts of the transition to carbon-neutral economy. The EU economy is currently significantly ramping up strategic efforts to safeguard an uninterrupted and geographical independent REE supply chain to support the sustainable energy transition.

A rapid and cost-effective method for screening discovery samples for REE content would enable more detailed resolution and/or broader survey area coverage. However, this would necessitate the daily analysis of hundreds or even thousands of samples. Depending on the rock/mineral composition, acid digestion may require various approaches and has implications for time and safety. Large mining companies within the North region have severely restricted for example the use of HF for digestions which favours in terms Li-tetraborate fusion as sample preparation.

The NexION 5000 ICP-MS from PerkinElmer, synchronized with the sample handling/ablation capabilities of LaserTRAX from Elemental Scientific Lasers, has made it possible to directly analyze solid samples via LA-ICP-MS in a high throughput environment. This work showcases the NexION 5000 Multi-Quadrupole ICP-MS as a sensitive and robust detector, suitable for the routine quantification of ultra-trace impurities and major components in solid matrices.

We present both short and long-term performance data of discovery samples in the determination of major and trace elements. Fused Li-tetraborate bead samples were first analyzed by XRF before being transferred to the LaserTRAX/NexION 5000 instrument. Automated data reduction was performed in Xceleri from Elemental Scientific Lasers, which synchronized sample IDs with quantitative data, regularly calibrated, and performed quality control checks.

(O-36)

Controlling sample washout

Thomas J. Kozikowski, Mike L. Booth and Autumn Phillips

Inorganic Ventures, Christiansburg, VA 24136 USA

E-mail: tkozikowski@inorganicventures.com

A lot of attention is given to spectral interferences when troubleshooting ICP analysis issues. However, memory interferences can be just as important. How can the analyst trust results if elements linger within the system for several subsequent samples? This presentation will focus on the specific cause for washout issues, identifying which elements to watch out for and why they can be troublesome. Specific chemical compatibility and acid matrix tables will be presented highlighting groups of elements that cause memory interferences. Introduction system design will also be discussed, and example data will be shown highlighting the effect of glass versus plastic components in the ICP as well as sample delivery method.

Several tips and tricks will be shared regarding how to mitigate sample washout. Recommended rinse solutions will be proposed based on sample contents as well as intro system selection. The differences between classic peri-pump sample delivery and switching valve applications will be emphasized and recommendations will be shared for both. Finally, some basic tips will be shared regarding ICP maintenance to emphasize the importance of frequent cleaning of introduction system components and how that will help control sample washout.

(O-37)

Automated analysis of low-to-high matrix environmental samples using ICP-MS with autodilution system

Andrew Brotherhood

Agilent Technologies, UK

ICP-MS is widely used in many environmental laboratories to quantify major and trace elements in low-to-high matrix samples such as waters, soils, and sediments. As these laboratories often need to analyze high numbers of varied samples every day, it is increasingly important to improve the efficiency of the workflow through automation.

To improve the efficiency of ICP-MS methods, a new inline autodilution system (ADS 2 by Agilent Technologies) has been developed that automates some time-consuming manual tasks. The autodilution system can be used to prepare calibration curves from a single stock solution, which significantly reduces preparation time and analyst workload. It can also automatically dilute samples before measurement, reducing preparation time of high matrix samples. Data quality can be enhanced through the automatic dilution and remeasurement of any samples where the concentration of target elements exceeds a user-defined concentration or internal standard elements exhibit suppression or enhancement. These features of the autodilution system alleviate the need for post-analysis rework, which reduces demands on the analyst and mitigates user-based errors.

Results from the measurement of a series of environmental reference materials by ICP-MS will be presented to demonstrate the accuracy and long-term stability of the autodilution method.

(O-38)

The need to meet: Mutual benefits for geochemists and chemists when discussing (LA)-ICP-MS/MS

Thomas Zack[1,2] and Sarah Gilbert[2]

[1] *Department of Earth Sciences, University of Gothenburg, Sweden*
[2] *Department of Earth Sciences, University of Adelaide, Australia*

The introduction of commercially available ICP-MS that incorporate a reaction cell sandwiched between two mass filters (called ICP-MS/MS) has placed a spotlight to ion-molecule reactions. For Earth Sciences, the realization that long lived beta decay schemes (e.g., ^{87}Rb decay to ^{87}Sr) can be utilized by using ICP-MS/MS is one of the most exciting discoveries in geochronology. Sample introduction via laser ablation (LA) allows it further to obtain age information from single crystals.

These developments have encouraged trial-and-error studies finding out what ion-molecule reactions optimize separation of daughter from parent isotopes (here separating $^{87}Sr^+$ from $^{87}Rb^+$). While most of those studies are building on decades of ion-molecule reaction studies, the focus on a handful of parent-daughter couples (namely ^{40}K and ^{40}Ca, ^{87}Rb and ^{87}Sr, ^{138}Ba and ^{138}La (^{138}Ce), ^{176}Lu and ^{176}Hf (^{176}Yb) and ^{187}Re and ^{187}Os) has exposed gaps in knowledge. For example, not all these elements are included in previous studies.

Potentially most exciting, recent studies (see Zack & Gilbert, 2024) has shown that better separation can be obtained by using gas mixes. Here, using a mix of N_2O and H_2 (or CH_4) is beneficial of separating $^{138}Ba^+$ from $^{138}La^+$ and $^{138}Ce^+$. Here, a wide range of important questions arises that require interdisciplinary collaboration: 1) a wide range of unusual reaction products are detected, what are they, 2) can gas mixes be modelled using density function theory and other theoretical tools, 3) what other gas mixes are useful (e.g., NH_3 and N_2O?).

(O-39)

Multiscale elemental analysis of materials using laser-assisted spectrochemistry

Andreas Limbeck, David Ken Gibbs, Maximilian Podsednik, Jakob Willner and Lukas Brunnbauer

TU Wien, Institute of Chemical Technologies and Analytics, Getreidemarkt 9/164-I²AC, 1060 Vienna, Austria

E-mail: andreas.limbeck@tuwien.ac.at

Laser-induced breakdown spectroscopy (LIBS) and Laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) are well established techniques for spatially resolved sample investigations. Attributes that make these two techniques attractive for imaging and depth profiling applications are their applicability to all kinds of solid samples, no or minimal sample preparation and fast sample throughput. While LIBS allows to determine almost every element of the periodic table, including elements such as H, C, N, O, and F, LA-ICP-MS offers better sensitivity down to the ng g−1 level, a prerequisite for measurements with increased resolution.

Since the signal generation of these analytical methods relies on firing a focused laser beam on the sample surface, they can be merged into a combined measurement method, where element specific information from one sample location can be acquired simultaneously by both techniques. Thereby, a more comprehensive sample characterization is enabled. However, to tackle the permanently rising demands in spatially resolved sample characterization, continuous development of this approach is inevitable.

In this contribution the development of a combined LA-ICP-MS & LIBS procedure is presented, which is based on measurement of single laser pulse responses with simultaneous LIBS and ICP-MS detection. Applicability of this approach with extended multi-element capabilities for fast mapping experiments with enhanced resolution is demonstrated by the analysis of selected samples from the field of material science.

(O-40)

Alternative approaches to calibration in LA-ICP-MS

Martin Šala

Department of Analytical Chemistry, National Institute of Chemistry, Hajdrihova 19, Ljubljana SI-1000, Slovenia

In recent years, significant progress has been made in enhancing the efficiency, sensitivity, and image clarity of LA-ICP-MS. Despite these advancements, precise quantification remains a challenge due to the difficulty in obtaining matrix-matched standards necessary for accurate calibration.

In our lab, we've devised two novel strategies to address this quantification issue in LA-ICP-MS. Leveraging the capabilities of ICP-TOFMS systems, which can measure all nuclides simultaneously, we've developed a semi-quantitative calibration protocol. This approach enables the measurement of nuclides even when specific standards are unavailable.

The key goal of using matrix-matched standards is to ensure that the amount of material ablated by the laser during analysis is accurately accounted for. Our alternative method involves correcting the signal based on the volume ablated, which is measured separately. This approach yields precise results even without matrix-matched standards, provided that adjustments are made for laser fluences.

Both of these approaches will be discussed, highlighting their advantages. We hope to demonstrate that we're making strides towards more universally applicable calibration methods in LA-ICP-MS.

(O-41)

LA-ICP-MS for critical raw materials characterization: Exploring the potential of secondary resources

Debora Foppiano, Sarina Bao, Casper Van der Eijk, Einar Jonsson and Anna Nordborg

SINTEF Industry, Sem Sælands vei 2 A, Trondheim, Norway

E-mail: debora.foppiano@sintef.no

Laser Ablation-Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS) is applicable to bulk analysis and mapping of critical elements along the various stages of the supply value chain, from raw materials (incl. recyclable materials) to highly refined products such as components in next-generation batteries.

Elemental content determination is typically done using techniques like atomic absorption spectrometry (AAS), inductively coupled plasma optical emission spectrometry (ICP-OES), and ICP-MS. These methods require converting solid samples into liquid solutions through processes like digestion, combustion, or fusion. The sample dissolution step has several drawbacks, including long preparation time and the risk of contamination or analyte loss. Most importantly, refractory minerals, (including zircon, garnet, or alumina) can withstand the dissolution process, reducing recovery rates for trace elements. Direct analysis of solid samples could overcome these issues and improve sensitivity by avoiding sample dilution.

The primary focus of this study was to quantify the presence of EU-listed critical raw materials in metallurgical slags and mine tailings. Determining whether these elements exist in sufficient quantities and concentrations is crucial for assessing their economic viability and potential recovery.

In this work we will present a comparison between ICP-MS/MS analysis on liquid samples obtained after microwave-assisted acid dissolution and LA-ICP-MS analysis for the determination of Mn ores and slags enriched in rare earth elements (REEs).

(O-42)

An approach to measure the number concentration and particle size distribution of microplastics using LA-sp-ICP-MS

Lukas Brunnbauer, Laura Kronlachner, Elias Foisner and Andreas Limbeck

TU Wien, Institute of Chemical Technologies and Analytics, Getreidemarkt 9/164-I^{2}AC, 1060 Vienna, Austria

Microplastics (MPs) are one of the major environmental challenges in our current times. To fully understand the impact of MPs on different ecosystem and to develop and employ appropriate regulations it is important to have analytical tools for a comprehensive characterization available. Nowadays, there is a wide range of analytical techniques used for the analysis of MPs each coming with their unique advantages and limitations. Besides conventional techniques such as optical microscopy, FT-IR and Raman spectroscopy also ICP-MS has found its way into the field of MPs. Several papers have been published reporting the use of single particle sp-ICP-MS to directly analyze the number concentration and size distribution in liquid samples. Additionally, a first work by van Acker et al. reported the use of LA-ICP-MS for a direct qualitative detection of individual MPs [1].

In this contribution, we propose a novel approach to prepare standards based on polymer thin films to quantify the size of the individual MPs in the low µm range enabling the investigation of the particle size distribution usingLA-sp-ICP-MS. Additionally, we investigate the transport efficiency (TE) of our LA-sp-ICP-MS method in respect to different measurement parameters, particle sizes and substrates. TE is an important metric in the field of sp-ICP-MS which is typically determined experimentally and directly influences the measurement of the particle number concentration.

Literature:
[1] Van Acker, Thibaut, et al. "Laser Ablation for Nondestructive Sampling of Microplastics in Single-Particle ICP-Mass Spectrometry." Analytical Chemistry 95.50 (2023): 18579-18586.

(O-43)

Pseudo-bulk elemental analysis of pegmatite lithium ores by LA-ICP-MS

Andrew M. Zipkin and Gideon Bartov

Eurofins EAG Laboratories, Liverpool, New York, USA

The lithium-ion battery supply chain is built on lithium-rich brine and “hard rock” lithium ore used for lithium carbonate and hydroxide production. The most important minerals for hard rock lithium are spodumene and petalite, which may co-occur in lithium-bearing pegmatite. The new work presented here uses LA-ICP-MS for major and minor element analysis of lithium ores and seeks to overcome the disadvantages of techniques that require digestion or fusion sample preparation. Typically, elemental analysis of lithium ore is accomplished by one or more of the following: sodium peroxide fusion paired with ICP-OES and/or ICP-MS, 4-acid digestion paired with ICP-OES and/or ICP-MS, and borate fusion paired with XRF. These techniques yield a bulk measurement of the ore. The major weakness of such approaches is sample preparation. Fusion and acid digestion are time-consuming, may lead to the loss of volatile elements, introduce opportunities for contamination, generate hazardous waste, and are destructive to the sample. LA-ICP-MS on pelletized ore powder mitigates the weaknesses of existing approaches but introduces a new challenge: it is a microanalytical technique that may not yield results representative of the bulk ore.

LA-ICP-MS is reliant upon matrix-matched calibrants that are homogeneous at the microanalytical scale. Nanopowders of rocks, sediments, and hard tissue have become increasingly available as microanalytical reference materials over the last decade. Here, we applied LA-ICP-MS to five lithium ore and ore concentrate reference materials that are commercially available from myStandards GmbH as nanopowder pellets. Using an ESL imageGEO 193 laser system coupled to a Thermo Scientific iCAP TQ ICP-MS, we explored combinations of calibrants before determining that a single point calibration using OREAS 999 ore concentrate (2.67 % Li) yielded the best results and most efficient instrument operation when measuring OREAS 750 – 753 for method validation. In addition to Li, analytes included Na, Mg, Al, Si, P, K, Ca, Ti, Mn, and Fe. Each secondary reference material was ablated 25 times over five days for a pseudo-bulk analysis. For OREAS 750-752 all analytes exhibited overall mean recovery within 10% of reference values, while OREAS 753 showed recovery within 25% of reference values for Mg, Ca, and Ti and within 10% for the other elements. Notably, data reduction in Iolite v4 relied upon the Si reference values for internal standardization.

Lastly, we analyzed a non-nanopowder lithium ore reference material, NCS DC 86303, as a pressed pellet made in-house. The certificate for NCS DC 86303 states only that particle size is <200 mesh (<75 µm); microscopy found aberrantly large discrete particles with maximum dimensions up to 400 µm. This non-microanalytical reference material was analyzed as a proxy for an unknown milled ore sample. Mean Si concentration for LA-ICP-MS internal standardization was determined by SEM-EDS at three locations on the pressed pellet since for an unknown sample a reference value would be unavailable. Based on ten replicate ablations, again calibrated with OREAS 999, mean recovery was within 25% of reference values for all analytes except Na and K, which yielded 133% and 137% recovery, respectively. With further development, matrix-matched, empirically calibrated LA-ICP-MS will be a promising alternative to existing approaches to lithium ore elemental analysis.

(O-44)

Method development and acquisition parameter optimization for single pulse resolved quadrupole LA-ICP-MS multielement analysis

Jakob Willner, Lukas Brunnbauer, David Gibbs, Maximilian Podsednik and Andreas Limbeck

TU Wien, Institute of Chemical Technologies and Analytics, Getreidemarkt 9/164-I^{2}AC, 1060 Vienna, Austria

In recent years, there have been significant developments in laser ablation technology. Ablation cells that enable fast aerosol washout in the millisecond range have been developed, which provides the possibility for high pixel acquisition rates in spatially resolved ICP-MS analyses. However, only ToF-ICP-MS instruments that cover the whole m/z range in each data point can exploit this benefit for fast and spatially resolved multielement analysis. With conventional quadrupole ICP-MS instruments, only one data point for each duty cycle is provided. Therefore, the spatial representation is typically calculated from transient quasi-continuous signals using the scan speed of the laser and predetermined aerosol washout times. However, using this conventional approach can lead to reduced image quality including imaging artifacts, such as pixel bleeding, image blur, or distortions. This is because the washout times may not be correctly determined or may change during the analysis of a heterogeneous sample.

To solve these issues, single pulse-resolved quadrupole ICP-MS acquisition is proposed. Achieving multielement analysis with a sequential quadrupole ICP-MS requires optimized settling and dwell times depending on the aerosol washout from single laser pulses. This work explores the basic requirements for successfully performing multielement analysis in single pulse-resolved quadrupole LA- ICP-MS measurements with fast washout cells. Washout times, transient signal durations, and acquisition parameters are systematically investigated to determine the multielement capability and limitations regarding the effect on accuracy and precision. The developed approach is applied on different micro-structured materials.

(O-45)

Impact of histological staining on the analysis of tissue thin sections using laser ablation-inductively coupled plasma-mass spectrometry

C. Verlemann[1], L. Schlautmann[1], M. Sperling[1], A. Radbruch[2], H. Richter[3], A. Jeibmann[4] and U. Karst[1]

[1]*University of Münster, Institute of Inorganic and Analytical Chemistry, Münster, Germany*
[2]*University Hospital Bonn, Clinic for Neuroradiology, Bonn, Germany*
[3]*University of Zurich, Diagnostic Imaging Research Unit, Clinic for Diagnostic Imaging, Department of Clinical Diagnostics and Services, Vetsuisse Faculty, Zurich, Switzerland*
[4]*University Hospital Münster, Institute of Neuropathology, Münster Germany*

Histological stains are used to differentiate and visualise relevant structures. The colouring of the components as well as its intensity help to assess and classify the patient's clinical status.
The distribution of endogenous and exogenous elements is important to better understand diseases or the pathway and effects of drugs in the body. Laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) is particularly suitable for the analysis of elements in tissue thin sections, because it is a sensitive method that allows to obtain a high spatial resolution of a few micrometres and has a wide linear range.

Usually, parallel sections are used for histological and element-specific examinations. potentially compromising comparability, particularly in case of small structures or if the tissue thin sections are not directly adjacent. Therefore, it would be advantageous to carry out both analyses on one section. Also, some stains contain elements like Cu or Fe, making them accessible to ICP-MS. Therefore, it would be possible to analyse the distribution of the stain by LA-ICP-MS to evaluate correlations of tissue types with endogenous and exogenous elements of interest.
In this study, the feasibility of a combined approach using ICP-MS compatible stains is investigated using three differently stained and one unstained tissue thin sections of the choroid plexus of a sheep previously administered a gadolinium-containing contrast agent for magnetic resonance imaging (MRI). The stains investigated were hematoxylin-eosin (HE), Elastica van Gieson (EvG) and Alcian blue/periodic acid-Schiff (AB-PAS). The entire plexus was analysed by LA-ICP-MS with a spot size of 8 µm and a smaller area with a spot size of 4 µm. All ICP-MS compatible stainings were detected at both spot sizes. Still, in comparison with the unstained thin section, washout behaviour was seen for some elements, possibly due to their different species, but especially for the gadolinium. So, the feasibility of doing the analysis by LA-ICP-MS on the stained thin section instead of an adjacent unstained thin section is depending on the staining procedure and the analytes of interest.

(O-46)

Single cell ICP-MS analysis: expanding biomedical applications

Maria Montes-Bayón, L. Gutierrez Romero, C. López Portugués, P. Díez García and M. Corte-Rodríguez.

Department of Physical and Analytical Chemistry. Faculty of Chemistry. University of Oviedo.
C/ Julián Clavería 8, 33006, Oviedo, Spain.
Health Research Institute of the Principality of Asturias (ISPA). Avda. Hospital Universitario s/n, 33011, Oviedo, Spain

The use of two-dimensional cell cultures to address the uptake of pharmaceuticals in biological systems is a first approximation to a rather complex problem such as drug delivery in tumor tissues. However, solid tumors are not a relatively homogenous mass of cells; they show complex architectures that contain necrotic regions, areas with cells in a quiescent state (growing slowly or not growing), and regions where cells proliferate rapidly. Thus, new in vitro biological models, which better represent the in vivo tissue, have been proposed leading to the development of the so-called multicellular tumor spheroids, organoids or patient-derived xenografts (PDX). All these models serve as an ideal choice in cancer treatment studies, such as preclinical trials of novel drugs, validating novel drug combinations, screening drug-sensitive patients, and exploring drug resistance mechanisms.

In this communication, the use of single cell ICP-MS experiments initiated in 2D cell cultures are extended to the evaluation of multicellular spheroids and furthermore, to the evaluation cells obtained from PDX. For this aim, new strategies for cell isolation and manipulation together with the use of ICP-MS measurement protocols need to be develop with the aim to compare Pt-based chemotherapeutic treatments outcome.

(O-47)

Molecular imaging utilizing laser ablation-atmospheric pressure chemical ionization-time of flight-mass spectrometry

J. Schmeinck, R. Brockington, L. Hansner, A. Kondrateva, T. Volkery and U. Karst

University of Münster, Institute of Inorganic and Analytical Chemistry, Münster, Germany

Postionization methods enable the hyphenation of laser ablation (LA) and molecular mass spectrometry. These techniques offer the potential to expand the scope of LA applications compared to laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) by preserving molecular sample information.1 Simultaneous analysis of analytes is made possible by time of flight-mass spectrometry (TOF-MS), a technique commonly employed for molecular analysis. These approaches also offer the advantage of enabling the imaging of native samples, addressing a primary limitation of matrix-assisted laser desorption/ionization (MALDI-MS).1 Atmospheric pressure chemical ionization (APCI) utilizes a corona discharge needle to create a plasma, which predominantly ionizes the carrier gas in a first reaction step. In a subsequent reaction cascade the analytes are chemically ionized, thus providing molecular information. This ionization source is well suited for the hyphenation to LA, as it is already a well-established method that is used to ionize the dried aerosol of liquid samples. Due to the original design for liquid samples, the development of a suitable sample introduction system and the optimization of various parameters is necessary to allow for higher analysis speed and higher signal intensities in molecular imaging.

Different designs for the sample introduction system into the APCI source were developed and tested regarding their impact on the achievable signal intensities and the washout behavior of the system. For each of the designs, the influence of the nebulizer gas, the dry gas and the gas flows of the LA system was evaluated. These optimizations were based on gelatine thin sections spiked with a selection of analytes, which were ablated in a standardized manner. Experiments were performed using a 213 nm laser ablation system coupled to a TOF-MS. Tablets sold as nutritional supplements, containing amino acids and vitamins, were used as samples for imaging experiments to determine the impact of these optimizations.

The optional nebulizer gas flow used to augment the sample flow was determined to lead to an inferior washout behavior and was therefore disregarded. Optimal settings were found for a sample introduction system that is able to direct the sample into the space in front of the corona discharge needle, where the plasma for the ionization of the analyte molecules is formed. Using this setup at low gas flows from the LA leads to faster washout of the analytes and much improved intensities. Most amino acids and vitamins in the tablets were identified based on their exact mass, showing various distinct distributions that matched with features visible on the surface of the tablet. Clear signs of slow washout behavior could be mitigated due to the optimized parameters, which allows for higher ablation speed and therefore faster analysis times.

[1] C. Herdering, O. Reifschneider, C. A.

(O-48)

Multimodal bioimaging of intracerebral hemorrhage by use of isotopically enriched blood in a mouse model by means of LA-ICP-MS and MALDI-MSI

Peter Niehaus[1], Raphael M. Buzzi[2], Kevin Akaret[3], Michael Hugelshofer[3], Dominik J. Schaer[2] and Uwe Karst[1]

[1]Institute of Inorganic and Analytical Chemistry, University of Münster, Münster, Germany
[2]Division of Internal Medicine, Universitätsspital and University of Zürich, Zürich, Switzerland
[3]Clinical Neuroscience Center, Universitätsspital und University of Zürich, Zürich, Switzerland

Intracerebral hemorrhage (ICH) represents a subtype of stroke that is characterized by the rupture of a cerebral vessel and subsequent bleeding into the brain tissue. While the initial bleeding causes immediate damage to the brain, the ICH-related secondary brain injury (ICH-SBI) is responsible for delayed neurological deficits. One of the reasons for ICH-SBI is thought to be the oxidative stress and neurotoxicity originating from released hemoglobin (Hb) and its metabolites. This is why investigating the fate of Hb-derived iron in affected brains as well as the occurrence of metabolites associated with oxidative stress will provide the basis for further elucidation of the mechanisms leading to SBI.

For bioimaging of the Fe distribution, laser ablation hyphenated to inductively coupled plasma mass spectrometry (LA-ICP-MS) was utilized. However, as an abundant endogenous element, iron displays a high background signal, impeding the localization of hemorrhage derived iron in the surrounding tissue. To circumvent this issue, in the presented mouse study, artificial ICH is induced by injection of ^{58}Fe-isotopically enriched mouse blood. The mice were sacrificed at different time intervals after injection. The brains were removed, snap frozen, and cryosectioned at 10µm thickness for bioimaging purposes. To investigate the impact of the hemorrhage on the tissue on a molecular level, matrix assisted laser desorption ionization imaging (MALDI-MSI) was utilized.

For LA-ICP-MS, ^{56}Fe and ^{58}Fe on brain sections were quantified using matrix-matched gelatin standards. Elemental bioimages were generated for the iron isotopes as well as for phosphorous, copper, and zinc to display anatomical features. Results show that the presented method provides high sensitivity for both iron isotopes as well as a clear differentiation between iron distribution caused by hemorrhage and natural background at a satisfactory spatial distribution. MALDI-MSI revealed complementary distributions of Hb and its metabolites, as well as a number of other biomarkers associated with oxidative stress in and around the bleeding site.

(O-49)

Development and characterization of nanoparticle-embedded inks for counterfeit detection using laser ablation single particle ICP-MS

T. Steinwachs[1], S. Baumann[2], T. Hirsch[2] and U. Karst[1]

[1]University of Münster, Institute of Inorganic and Analytical Chemistry, Corrensstr. 48, 48149 Münster, Germany
[2]University of Regensburg, Institute of Analytical Chemistry, Chemo- and Biosensors, Universitätsstr. 31, 93053 Regensburg, Germany

Counterfeiting is a major global challenge that affects virtually every industry, with consequences ranging from financial loss to potential health risks. Product labelling is therefore an important means of verifying the authenticity and safety of products. One possibility for labelling is the use of nanoparticles, which can be clearly identified by various characteristics such as their magnetic or optical properties or elemental composition. The use of lanthanide-based upconversion nanoparticles (UCNPs), which are capable of converting multiple low-energy photons into a single higher-energy photon, offers a combination of labelling properties. Fluorescence, elemental composition and different stoichiometries of the 15 lanthanides can be used as (individual) labels. Such labels can be decoded using inorganic mass spectrometry techniques such as ICP-MS. In particular, the use of single particle ICP-MS (spICP-MS) allows the characterization of UCNPs in terms of size and concentration in addition to elemental composition.

A range of carriers, including commercially available printer and writing inks, along with a custom hydrophilic ink based on water-ethanol, glycerol and sodium dodecyl sulfate was investigated. Citrate-stabilized gold nanoparticles (AuNPs) and UCNPs were added to the inks. The surface of the UCNPs was modified with polyacrylic acid improving their dispersion in aqueous ink solutions.

The labelled inks were tested by printing and applying them to paper and glass slides, followed by analysis using laser ablation with spICP-MS (LA-spICP-MS) detection. The elemental composition and size distribution of the embedded nanoparticles were determined by optimizing various parameters, such as pixel size, ablation speed and laser energy. Additionally, the influence of different matrices on the analysis and stability of the nanoparticles was considered. Furthermore, to demonstrate the ability to spatially distinguish between different nanoparticles, the university's logo was printed using nanoparticle-labelled inks and the analyzed by LA-spICP-MS.

(O-50)

Single cell – inductively coupled plasma – mass spectrometry (SC-ICP-MS): A powerful tool to study metals during host-pathogen cell interactions

Claire Davison[1,2], Jordan Pascoe[2], Melanie Bailey[1], Dany JV Beste[2] and Mónica Felipe-Sotelo[1]

[1]School of Chemistry and Chemical Engineering, Faculty of Engineering and Physical Sciences, University of Surrey, Guildford, UK [2] Department of Microbial Science, Faculty of Health and Medical Sciences, University of Surrey, Guildford, UK
E-mail:claire.davison@surrey.ac.uk

Single cell – inductively coupled plasma – mass spectrometry (SC-ICP-MS) offers an attractive option for rapidly measuring trace metal heterogeneity at the single cell level. Chemical fixation has been previously applied to mammalian cells prior to sample introduction so that they can be resuspended in a solvent suitable for SC-ICP-MS. However, the effect of fixation on the elemental composition of suspended cells is unknown. We have developed a novel strategy to compare cell suspensions treated using different fixatives (methanol 60-100% in H_2O) and 4% paraformaldehyde (PFA) in phosphate buffered saline solution (PBS), based on the number of distinguishable single cell events, keeping a constant particle number concentration. We demonstrated that different fixatives can have a significant impact on measured cell elemental composition.

Trace metals are essential for intracellular pathogens to survive within their hosts. However, host cells also use these metals as bacteriostatic/bactericidal weapons and therefore, understanding metal homeostasis could lead to novel therapeutics such as micronutrient therapies. We have also shown for the first time that SC-ICP-MS measurements of infected THP-1 macrophages is possible by applying our methodology (see Figure 1) to investigate the effects of mycobacterial infection on the elemental composition of human macrophages, demonstrating its utility in understanding metal homeostasis during microbial infection. Whilst there was no significant difference in the average mass of magnesium, calcium and zinc per THP-1 macrophage (n=3, $P \leq 0.05$) the work identified significant cellular heterogeneity across cell populations that would not be seen in bulk measurements.

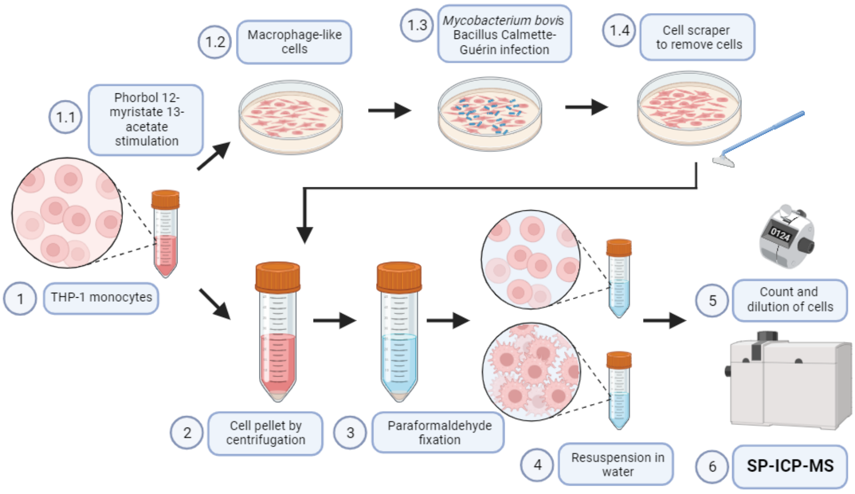


Figure 1 Sample preparation procedure used for the measurement of trace metals in single THP-1 monocytes, non-infected macrophages and BCG infected macrophages using SP-ICP-MS.

(O-51)

Multi-element/isotope single-cell analysis by ICP-time of flight (TOF)-MS

Xiangwei Tian[1], Yongguang Yin[1], Yong Cai[2] and Guibin Jiang[1]

Research Center for Eco-Environmental Sciences, Chinese Academy of Sciences, Beijing 100085, China
Department of Chemistry and Biochemistry, Florida International University, Miami, Florida 33199, United States
E-mail: ygyin@rcees.ac.cn; cai@fiu.edu.cn

Direct detection of chemical elements/isotopes on cells commonly relies on digestion-based bulk analysis. However, the heterogeneity in elemental/isotopic distribution due to diverse cellular physicochemical and physiological properties is submerged into the population-averaged results. The challenge has been addressed by single-cell inductively coupled plasma mass spectrometry (scICP-MS) for high-throughput and high-sensitivity quantification of the analyte elements/isotopes on individual cells. Moreover, emerging techniques for multi-element/isotope detection can provide multi-dimensional information at the single-cell level.[1]

The multi-element distribution profiles for human sperm cells recorded by scICP-time of flight (TOF)-MS revealed varied degrees of heterogeneity for different elements. Endogenous elements with high abundance, such as P and Zn, showed relatively low heterogeneity.[2] Algal accumulation experiments revealed differences in heterogeneity for the accumulation of isotopically labeled exogenous $^{199}\mathrm{Hg}^{2+}$ and ^{201}MeHg. Within the same algae population, a higher degree of heterogeneity was observed in cellular MeHg contents with a relatively higher abundance. The varied degrees of heterogeneity among the analytes further highlight the necessity of multi-element/isotope single-cell analysis. Another important application of multi-element single-cell analysis is label-free cell identification. The reliability of scICP-MS data can be improved by using simultaneously detected endogenous elements as cell indicators. For example, when detecting algae cells exposed to Ag nanoparticles, false-positive cell events attributed to free Ag nanoparticles can be excluded according to the absence of simultaneously detected endogenous P signals.[3]

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(O-52)

Single particle TOF-ICP-MS for the determination of atmospheric particles

Yannick Tardif, Katia Iatariene, Houssame-Eddine Ahabchane, Madjid Hadioui, Patrick Hayes and Kevin J. Wilkinson

Biophysical environmental chemistry, University of Montreal, Campus MIL, 1375 Ave. Thérèse-Lavoie-Roux, Montréal, QC, H2V0B3, Canada

Elevated concentrations of fine ($PM_{2.5}$) and ultrafine particulate matter are associated with poor air quality and a variety of adverse health effects. While single particle techniques have the advantage of being able to provide information on the occurrence and nature of low frequency events, techniques to measure the chemical composition of $PM_{2.5}$ on a single particle level are limited. In this work, particles were collected using filtration or a Coriolis sampler. Single particle inductively coupled plasma time-of-flight mass spectrometry (SP ICP-ToF-MS) was used to compare the composition of aerosol particles collected from pristine, urban and industrial settings.

Comparisons of chemical compositions analysed on a particle-by-particle basis provided us with insight into the nature of the risk associated with the particles. Insight into the limitations due to sampling (collection of particles), analysis (detection limits, multiple dilutions, optimal particle numbers) and data treatment (data filtering, cluster analysis) will be discussed. Sampling with a Coriolis sampler ultimately was found to be the most thorough for the sampling of the smallest particles. Particle size detection limits were lowered by using a combination of ion-exchange resins and oversampling (limiting of mass ranges analysed). Hierarchical cluster analysis provided limited insight into differences among the samples. Rather, clustering, based upon elemental composition only, was more useful for distinguishing samples and identifying markers of atmospheric pollution.

Finally, for samples collected from several contrasting sample locations (Northern Canada, urban Montreal, industrial sample, etc.), SP ICP-ToF-MS analysis showed that a detailed analysis of the low frequency events (~<3%) provided more insight into the nature of the sample as opposed to high frequency events that were largely similar among sites. For example, by focusing on Ba, Rb, Cr, Pb, Ni, Zn, Cu and Ag, the urban and Northern samples could be distinguished, and the anthropogenic contribution estimated.

(O-53)

Characterisation of small microplastics by spICP-MS: Dynamic mass flow method for the SI traceable quantification of number-concentration

D. Ojeda, A. Sikora, D. Bartczak and H. Goenaga-Infante

LGC National Measurement Laboratory, Queens Road, Teddington, UK

E-mail: david.ojeda@lgcgroup.com

Plastic pollution has been widely recognised as a global environmental problem. Two-thirds of the 359 million tonnes of plastic produced annually at the global scale is released into the environment, where it undergoes complex physicochemical processes, degrading over time. Larger pieces of plastics are fragmented into small microplastics (SMPs). SMP in turn accumulate in the environment, especially in water and soil, then enter food chain; however, the potential risks from exposure to smaller plastic particles in humans are yet to be fully understood. In this context, information regarding characteristics and number-based quantities for small microplastics (SMPs, 1-10 µm) is needed for a comprehensive risk assessment. Such assessments for SMPs are required since particles at the lower end of the microscale pose the biggest threat to human health and the environment.

Significant progress has been made in recent years with regards to the development of analytical methods for identification and size characterisation of SMPs. Single particle ICP-MS (spICP-MS) has emerged as a promising technique for their number-based quantification. However, there are remaining challenges including the lack of reference materials for method validation[1] as well as the combined high C isotopes background signal with insufficient sensitivity for their detection at very low concentrations in real samples.[2,3]

This lecture will discuss the potential of spICP-MS with the Dynamic Mass Flow (DMF) approach for number concentration value assignment to SMPs, with direct traceability to the SI unit of kg. The suitability of DMF for number-based determinations of SMPs will be demonstrated and its feasibility for the characterization of microplastics with a size range of 1-10 µm will be discussed. Confirmatory data obtained with an orthogonal approach based on optical detection using an XPT flow particle analyzer will also be highlighted. Finally, challenges related to TE determination for the larger size end will be discussed.

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(O-54)

Dynamic mass flow approach for the accurate determination of particle number concentration

Armando Sánchez-Cachero, David Ojeda, Kharmen Billimoria, Dorota Bartczak, and Heidi Goenaga-Infante

LGC National Measurement Laboratory, Queens Road, Teddington, Middlesex, TW11 0LY, United Kingdom

E-mail: armando.cachero@lgcgroup.com

During the past five years, single particle inductively coupled plasma mass spectrometry (spICP-MS) has gained popularity for the characterization of nanoparticles (NPs) due to its unique features to provide direct measure of size-resolved particle number-concentration (PNC) and the dissolved element mass fraction simultaneously. PNC is one of the key parameters required for the characterisation of nanomaterials in line with the European Union legislation and guidelines [1, 2]. Several approaches have been described to determine transport efficiency which is essential for PNC determination using spICP-MS [3]. The most widely used methods are reliant on reference materials which are limited or unavailable. Recently, the Dynamic Mass Flow (DMF) approach was proposed to provide accurate data for nanoparticle number concentration under specified optimal operating set up and conditions [4] without the need for calibration with reference materials. The main use of this method has been highlighted as being invaluable for the assignment of a SI traceable number concentration value to new, commercial nanomaterials and use those as quality control materials in spICP-MS experiments.

This work describes a comprehensive study aiming to obtain further insights into the feasibility of the DMF method to obtain accurate PNC under operating ranges and set ups outside of the published recommendations [4] but that still comply with key method requirements (e.g. use of an ICP-MS system in equilibrium, a cooled spray chamber., etc). In this vein, different types of sample introduction systems, ICP-MS instrumentation, spray chamber temperatures and sample uptake flowrates were investigated using 60 nm citrate capped AuNPs, and 75 nm AgNPs (NIST 8017) as quality control materials. Best practice recommendations for the correct implementation of the DMF approach e.g. in terms of key measures of ICP-MS stabilisation time, batch sequence specifications, or sample composition and stability requirements will also be discussed.

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(O-55)

Advantages of single-cell ICP MS to monitor the metal uptake by green algae

Julia Dressler[1], Muhammad Younas[2], Alexander Köhrer[1], Matthias Elinkmann[1], Derrick Quarles Jr[3], Michael Hippler[2] and Uwe Karst[1]

[1] *University of Muenster, Corrensstraße 48, 48149 Muenster, Germany*
[2] *University of Muenster, Institute of Biology and Biotechnology of Plants, Schlossplatz 8, 48143 Münster, Germany*
[3] *Elemental Scientific, Atlanta, GA & Omaha, NE, USA*

Inductively coupled plasma mass spectrometry (ICP-MS) has been established as a reliable analytical technique for the analysis of the elemental content in biological systems. The total metal concentration in cells can simply be determined by cell lysis, homogenization, and subsequent analysis via ICP-MS. [1] However, by destroying the cells and homogenizing the sample, only average metal contents are determined over the entire cell population, while information about the heterogeneity of the cell population is lost. It is therefore of interest to use methods that allow the analysis of individual cells. In the past years single cell-ICP-MS (SC-ICP-MS) has been established as a promising technique for the elemental analysis of individual cells. [2]

The present study shows that SC-ICP-MS can resolve the heterogeneity of a cell population and thus provides insight into different uptake behavior of metals by cells. For this purpose, *Chlamydomonas reinhardtii*, a species of green algae, were incubated with nickel under two conditions: (A) nutrient-poor conditions and (B) nutrient-rich conditions. Quantitative SC-ICP-MS analysis was performed using a PlasmaQuant MS / Elite equipped with a total-consumption-on-axis spray chamber and a microFAST SC autosampler. 60Ni+ and 24Mg+ were monitored. Magnesium is an endogenously occurring element in these algae, which serves as a reference. Additionally, lysis and direct injection ICP-MS was performed for comparison purposes.

In both incubation experiments, significant nickel uptake was observed after 24 hours of incubation, while after 48 hours, the nickel content in the cells decreased again. This was detected using direct injection ICP-MS and SC-ICP-MS. However, SC-ICP-MS enables the possibility to resolve variations between the two experiments. In experiment (A), a homogeneous nickel uptake of 5 fg/cell was observed, while in experiment (B), a heterogenous uptake was detected. In this case, two distinct populations were detected with a nickel content of 2 and 11 fg/cell. The cell population's heterogeneity is also reflected in the magnesium content. It can also be observed that the cells from experiment (A) have a significantly lower magnesium content after just 24 hours of incubation. After 48 hours, a very low magnesium content per cell is detected in both experiments. This indicates that the loss of magnesium could possibly be a stress reaction of the cells to the exogenous nickel.

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(O-56)

Strategies for the reliable quantification of mass and number concentration of nanomaterials in food: The power of a multimethod approach centered around ICP-MS

D. Bartczak[1], D. Ojeda[1], Zoltán Varga[2], and H. Goenaga-Infante[1]

[1]*UK National Measurement Laboratory (NML) at LGC Limited, Queens Road, Teddington, UK*
[2]*Institute of Materials and Environmental Chemistry, HUN-REN Research Centre for Natural Sciences, H-1117 Budapest, Magyar Tudósok körútja 2., Hungary*

E-mail: dorota.bartczak@lgcgroup.com

Food additives are widely used to improve the production, quality, and durability of foods. However, certain food additives have recently raised safety concerns due to the presence of nanomaterials (NM). Although NM themselves offer a potential new food technology they require authorization under the requirements of the Novel Foods Regulation 2015/2283, and their presence in food must be indicated on the label (EC Regulation 1169/2011). European legislation is underpinned by the Recommendation 2022/C 229/01 for the definition of NM, which describes the need for reliable identification of NM and determination of a percent content of nanoscale particles within a consumer product or an additive. However, NM characterization in a regulatory or monitoring context, especially at the lower end of the nanoscale, is challenging due to the multimodal, non-spherical and multi-elemental character of ‘real-life’ NM added to food combined with the high complexity of the food matrix. Therefore, recent research efforts have been focused on a development and validation of reliable measurements methods for the quantification and characterization of NM in food to support food industry, reference and testing laboratories as well as monitoring agencies to comply with emerging regulations.

To this end, ICP-MS hyphenated platforms have been demonstrated to be powerful for the detection and reliable quantification of mass and number concentration of inorganic NM in food. However, there are remaining analytical challenges associated with reliable mass and number-based quantification of NM at the lower end of the nanoscale.

This lecture will discuss how some of those challenges have been overcome through (*i*) development and application of a platform based on Single particle ICP-MS (spICP-MS), supported by asymmetrical flow field-flow fractionation (AF4) coupled on-line to ICP-MS and multi-angle light scattering (MALS) to the identification and number-based quantification of TiO_2 particles with sizes down to 15 nm, in the raw ingredient TiO_2 (E171) as well as in a range of food products[1] and (*ii*) use of AF4/ICP-MS with size fraction-targeted isotope dilution analysis (IDA) for the reliable detection and quantification of nanoscale silica (SiO_2, E551) mass fraction in the raw additive and food samples.[2] To achieve this, synthesis and characterization of 29Si-enriched SiO_2 nanoparticle spike/internal standard was undertaken and the associated advantages and drawbacks will also be discussed.[3] The lecture will finally discuss on the power of combined AF4/ICP-MS with off-line Transmission Electron Microscopy (TEM) for the simultaneous size-resolved mass determination and identification of multiple NM additives in food.

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(O-57)

Online elemental analysis in hydrometallurgical production and recycling processes of lithium-ion battery metals using micro-discharge optical emission spectroscopy

Toni Laurila and Aappo Roos

Sensmet Ltd, Finland

Both in lithium-ion battery metal production and recycling there is a growing need for new fast, precise and easy-to-use analytical techniques, especially for the online process monitoring. Established techniques of ICP-OES and ICP-MS are the workhorses in the laboratories for metals analysis, yet they are off-line making them unsuitable for real-time industrial process monitoring. Therefore, a fast and precise technique that allows online metal analysis would be preferable. Here we report on automated online quantification of battery metals in hydrometallurgical processes using novel micro-discharge optical emission spectroscopy (μDOES). The technology is based on a micro-plasma which is directly created inside an aqueous sample without any carrier gas by using electrodes and high voltage electrical pulses. The μDOES analyser design is simple and robust having no high-maintenance components in continuous 24/7 operation.

In this contribution we present the analytical performance of micro-discharge optical emission spectroscopy in real-time quantification of both major battery metals [1] as well as impurity metals [2] in hydrometallurgical processes. The applicability of the technique to online analysis of hydrometallurgical black mass from recycled lithium-ion batteries is also covered. The results show that micro-discharge optical emission spectroscopy can reach sub-µg/L limit or detection and 1-2% repeatability in online metals analysis. Furthermore, we demonstrate how the technique can perform extremely fast online metals detection from process liquid streams with a response of time of one second which is beneficial for direct lithium extraction processes, for example.

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(O-58)

Challenges in the determination of toxic elements in food additives

Daniela Montalvo Grijalva[1], Céleste Vansnick[1], Noa Olluyn[1], Iris Wyns[1], Jan Mast[1], Nadia Waegeneers[2] and Karlien Cheyns[1]

[1]Sciensano, Unit trace elements, Tervuren, Belgium
[2]Sciensano, Unit Risk and Health Impact Assessment, Brussels, Belgium
E-mail: Karlien.Cheyns@sciensano.be

In several opinions on the re-evaluation of food additives (FAs), EFSA's Food additives and Flavourings (FAF) Panel concluded that the potential exposure to several toxic elements (e.g. arsenic (As), lead (Pb), cadmium (Cd), mercury (Hg)) resulting from the consumption of these additives could be significant. Consequently, in recent published and ongoing re-evaluations (and follow-ups) of several food additives, the FAF Panel recommends that the current limits for toxic elements be lowered and that missing limits (e.g. aluminium (Al)) be included in their specifications.

The determination of the toxic elements in pristine FAs poses specific analytical challenges, as the analytical methods commonly used for food products, based on acid digestion and measurement by ICP-MS or ICP-OES, are not transferable *as such* for several FAs. Therefore, method optimisation and validation were performed for As, Cd, Pb, Al in a wide range of FAs. The food additives were selected on the basis of their use levels and impurity specifications. Measurements were performed by ICP-MS/MS and ICP-OES.

Organic synthetised FAs do not necessarily interfere with the measurement of the toxic elements, but e.g. glycerol and fat or oil derived food additives can cause explosive reactions during mineralisation.

The analysis of impurities in mineral FAs is even more challenging. Calcium (Ca) and sodium (Na) carbonates (E170, E500) and other Ca or Na salts, cause signal suppression in the plasma. In the case of iron oxide (E172), high temperature and pressure are required for complete digestion and the determination of Al by ICP-OES can be interfered by the presence of Fe. Handling of the pristine product silica (E551) requires the use of deionisers and complete mineralisation requires the use of harmful hydrogen fluoride. For this FA, in addition to a method for complete mineralization, method validation is also performed after HCl extraction. The latter method results in a more realistic bioavailable fraction but a challenging matrix for ICP-MS analysis.

For validation purposes, FAs were grouped for chemical comparability and a representative FA was spiked under reproducible and repeatable conditions. As the presence of the matrix affects the limit of quantification (LOQ), the LOQ was determined in a representative matrix. As no certified reference material is available, the trueness was estimated from the spikes. Results of the method validations and tips and tricks are presented.

(O-59)

Investigating element concentrations in water samples from the West Indian Ocean

Michaela Zeiner[a], Wolfgang Kandler[b] and Heidelore Fiedler[a]

[a] *Örebro University, School of Science and Technology, MTM Research Centre, Fakultetsgatan 1, 701 82 Örebro, Sweden*
[b] *University of Natural Resources and Life Sciences, Vienna, Department of Agrobiotechnology, IFA-Tulln, Institute of Bioanalytics and Agro-Metabolomics, Konrad-Lorenz-Str. 20, 3430 Tulln, Austria*

The concentrations of chemical elements dissolved in seawater cover a range of far more than ten orders of magnitude due to natural abundance and solubility. Regarding to their mass concentrations, Cl, Na, Mg, S, Ca, and K (in this order) normally cover 99,95% of the dissolved matter. Trace elements are present in lower concentrations, but can play important roles in many biogeochemical processes, including nutrient cycling and the formation of marine sediments. Some of them can be highly toxic to marine organisms and may bioaccumulate in the food chain. Simultaneous multielement determination is challenging and requires analytical methods with high dynamic range. Inductively coupled plasma mass spectrometry (ICP-MS) gives the possibility to quantify all major elements and about 20 trace and ultra-trace elements in seawater at natural concentration levels without time-consuming sample pre-treatment. The samples were diluted 1:60 and the external calibration was based on matrix matched standard solutions.

The method was optimised for the accurate determination of major and trace elements in seawater samples, such as off the coast of Tanzania and Mozambique in the West Indian Ocean (WIO). This water body is warmer than many other aquatic systems and characterized by high salinity. Surface water as well as deep-water samples were analysed to provide a snapshot as to the occurrence of 42 elements in a water body at low latitudes, with monsoonal winds and reversing currents.

Fourteen of the 42 elements determined were below the limits of quantification in most of the samples. The results of the remaining elements were assessed based on sampling site (location), depth, pressure, salinity, oxygen content, conductivity, and water temperature. Only for Si and P changes in concentrations with depth (or pressure) were observed, having correlation coefficients of R=0.94 and R=0.97. The increases with depth were more pronounced in the samples from Mozambique (greater depths) than Tanzania. Conductivity, temperature, and dissolved oxygen decreased with depth (vertical distance from the water surface) and pressure.

Some alkali and alkaline earth metals, namely Li, Na, K, Rb, Mg, Ca, but also U and V were highly correlated with Cl and B but also with S.

A comparison of 18 elements determined in plankton from the same locations (van Aswegen, 2020) and in the water samples revealed very distinct patterns: whereas the plankton samples were dominated by Ca, followed by Mg, and Sr, the order in the water samples was Mg >> Ca > K.

Van Aswegen, J. D. (2020). *Metal composition of zooplankton from the Western Indian Ocean* (Doctoral dissertation, North-West University (South Africa))

(O-60)

Accurate arsenic determination in pyrolysis oils using a total sample introduction system in combination with ICP-MS/MS

Raquel Sánchez,[a] Mar Todolí,[a] Rebeca Pérez,[a] Thomas Coquet,[b] Marion Lacoue-Nègre,[b] and Fabien Chainet[b]

[a] *Department of Analytical Chemistry, Nutrition and Food Sciences, P.O. Box 99, 03080, Alicante, Spain*
[b] *IFP Energies nouvelles, Rond-point de l'échangeur de Solaize, BP3, 69360 Solaize, France*

The rigorous monitoring of metals and metalloids, such as arsenic, in pyrolysis oil samples is crucial for several reasons: *(i)* these elements can influence the stability of fuels and their precursors; *(ii)* due to its toxicity, arsenic should not be released into the environment; *(iii)* arsenic could lead to the severe deactivation of the active sites of catalysts used in hydroprocessing steps; and *(iv)* some elements may serve as additives, for example arsenic is used as bactericide. Due to its high sensitivity and low detection limits, Inductively Coupled Plasma Mass Spectrometry (ICP-MS) is recommended for arsenic determination in these organic matrices. However, introducing organic samples into the spectrometer may cause problems such as plasma thermal degradation, spectral interferences, and soot deposition on the ICP-MS torch or interface cones, which are typically mitigated by oxygen stream addition. ICP-MS/MS helps to reduce spectral interferences, crucial for accurately quantifying arsenic. For example, interfering ions, such as $^{40}Ar^{35}Cl^+$, $^{38}Ar^{37}Cl^+$, $^{59}Co^{16}O^+$, $^{40}Ca^{35}Cl^+$, Sm^{2+} and $^{150}Nd^{2+}$ could overlap with $^{75}As^+$ peaks. The complex matrix of pyrolysis oil samples could influence the analytical process from nebulization to ionization, leading to matrix effects. Additionally, the arsenic chemical form could affect the sensitivity, thus degrading the accuracy of the determination.

Results obtained in the present work have demonstrated the feasibility of ICP-MS/MS for accurately determining arsenic concentrations in complex matrices. The methodology involved diluting the sample in xylene, applying a 10-dilution factor, followed by signal measurement using the so-called high temperature torch integrated sample introduction system (hTISIS) working at 400°C, combined with ICP-MS/MS. The hTISIS system significantly enhanced analytical performance, providing an order of magnitude better sensitivity and improved detection limits as compared to the conventional sample introduction system. The combination of hTISIS and ICP-MS/MS allowed for the mitigation of matrix and chemical form effects, achieved through the use of high-temperature conditions, and spectroscopic interferences, due to the introduction of oxygen into the spectrometer as a reaction gas. Consequently, the universal calibration approach could be applied. An external calibration method with a single set of standards prepared in a suitable solvent like xylene was feasible for analyzing pyrolysis oil samples.

(O-61)

Use of ICPMS in official food and feed control – experiences from the European Union reference laboratory (EURL-MN)

Jens J. Sloth, Katrin Löschner, Lucas Givelet and Heidi Amlund

National Food Institute, Technical University of Denmark, Kemitorvet B201, DK-2800 Kgs. Lyngby, Denmark and the European Union reference laboratory for metals and nitrogenous compounds (EURL-MN)

The National Food Institute at the Technical University of Denmark has since 2018[1] hosted the European Union reference laboratory for metals and nitrogenous compounds in feed and food (EURL-MN). The EURL-MN succeed the former EURLs in the area: EURL-HM at JRC-IRMM (Heavy metals in feed and food) and EURL-CEFAO at ISS in Italy (Chemical elements in food of animal origin). The scope of the new EURL was extended to include all metals –and nitrogenous compounds was added as a new group of compounds. The EURL leads a network of national reference laboratories (NRLs) in EU, EFTA and EU candidate countries and the network comprises currently 60+ laboratories.

The role of the EURL-MN is to contribute to the improvement and harmonisation of analytical methods used for official feed and food control. The activities cover both the regulated[2, 3] compounds (e.g. arsenic (incl inorganic arsenic), cadmium, lead, mercury, nitrite and nitrite) as well as compounds of monitoring and/or potential future regulatory interest (e.g. aluminium, nickel and N-nitrosamines).

In this presentation a general introduction to food and feed control scene in EU will be provided and the role and activities of the EURL-MN presented. The talk will focus on observations and experiences from the EURL-MN with regards to the use of ICPMS in official food and feed control.

[1] Commission Regulation (EU) 2018/192 amending Annex VII to Regulation (EC) No 882/2004 of the European Parliament and of the Council as regards the EU reference laboratories in the field of contaminants in feed and food.

[2] Commission Directive 2002/32/EC on undesirable substances in animal feed.

[3] Commission Regulation (EC) 915/2023 on maximum levels for certain contaminants in food and repealing Regulation (EC) No 1881/2006.

(O-62)

Plasma techniques for the analysis of lithium ion batteries

Dennis Kessen[a], Tobias Brake[a], Marc Vahnstiege[a], Malina Helling[a], Kai Brüning[a], Till Kröger[a], Martin Winter[a,b], Sascha Nowak[a] and Simon Wiemers-Meyer[a]

a MEET Battery Research Center, Corrensstr. 46, 48149 Münster, Germany
b Helmholtz-Institute Münster, IEK-12, Forschungszentrum Jülich GmbH, Corrensstr. 46, 48149 Münster, Germany

Global lithium ion battery production is rising steadily, which means that the number of batteries in circulation is also increasing. At the same time, more and more research is being carried out to further develop current battery technology in order to further improve criteria such as energy density, power, safety and sustainability. As a result, the need for reliable analysis methods for characterization of battery materials and battery cells is becoming ever greater. Suitable analytical methods are not only required for quality control of battery cell manufacturers but also private and public research institutes, analytical service companies and OEMs have to be able to analyze such samples. Without analytical chemistry batteries remain black boxes.

The analysis of lithium ion battery samples, however, can be challenging, as the cells contain a broad variety of different substance classes with many highly reactive compounds. Regarding elemental analysis with plasma-based techniques the challenges can be related to the determination of spatial distributions and simultaneous determination of high and low concentrations. However, plasma-based techniques offer many possibilities to analyze battery samples, especially when quantitative results are required.

This presentation will give an overview of applications of plasma-based analysis techniques in the field of lithium ion batteries. It will cover capabilities and challenges of routine and research techniques, such as, ICP-OES, ICP-(SF-)MS and ETV-ICP-OES to determine elemental concentrations in battery materials, LA-ICP-MS and GD-MS to perform spatially and depth resolved measurements, single-particle ICP-OES to investigate the state of charge of individual active battery material particles as well as GC-BID to analyze gaseous side products formed during battery operation.

13 Short Course Abstracts

(A-1)
A brief introduction to LIBS

Vincent Motto-Ros

Institut Lumière Matière, iLM UMR5306 Université Lyon 1-CNRS, 69622 Villeurbanne, France.
E-mail : vincent.motto-ros@univ-lyon1.fr

In recent years, spurred by the achievements of NASA's Martian exploration missions, Laser-Induced Breakdown Spectroscopy (LIBS) has undergone remarkable advancements. These advancements encompass the development and commercialization of highly efficient portable LIBS instruments [1], alongside the establishment of LIBS imaging as a benchmark elemental imaging technique [2,3]. In LIBS, the application of laser pulses to the surface of a sample induces a plasma, from which elements within the sample emit a distinct optical response (see Figure 1). This response stems from the electronic relaxation of excited atoms and ions, prompted by the intense plasma temperature, and is subsequently captured and analyzed using an optical spectrometer.

In this course, we will elucidate the essential facets necessary for achieving successful LIBS measurements. This encompasses a comprehensive understanding of plasma physics, instrumentation, data analysis, and processing. While the generation of laser-induced plasma is relatively straightforward, ensuring its reproducibility in practical settings presents significant challenges. Consequently, our focus will be on optimizing LIBS instrumentation and exercising precise control over experimental parameters. Through illustrative examples, we will delineate both the merits and limitations of this technique. Finally, we will conclude with an exploration of LIBS quantification methodologies and an introduction to LIBS imaging techniques.

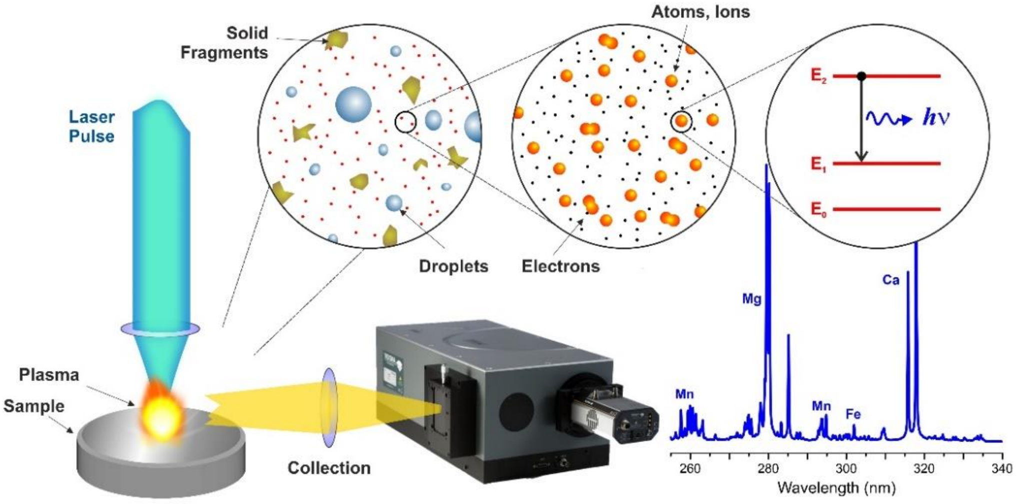


Figure 1. LIBS principle.

References:
[1] R. S. Harmon, R. E. Russo, R. R. Hark, Spectrochim. Acta, Part B 87, 11 (2013).
[2] B. Busser, S. Moncayo, J.-L. Coll, L. Sancey, V. Motto-Ros, Coordination Chemistry Reviews 358, 70-79 (2018).
[3] V. Gardette, V. Motto-Ros, C. Alvarez-Llamas, *et al.*, Analytical Chemistry 95, 49-69 (2023).

(A-2)

Quality of measurements: Basics of method validation, traceability and uncertainty assessment

Johanna Irrgeher

Montanuniversität Leoben, Chair of General and Analytical Chemistry, 8700 Leoben, Austria

E-mail: johanna.irrgeher@unileoben.ac.at

This course offers an introduction to the principles of (1) method validation, (2) traceability, and (3) the assessment of measurement uncertainty, fundamental to ensuring the quality of analysis. Participants will get an introduction to the basic concepts of metrology in chemistry with a focus on method validation including calibration procedures, thereby enhancing the validity and reliability of their data.

The concept of traceability of measurements is achieved by establishing an unbroken chain of comparisons to national or international standards. This framework provides confidence in the accuracy and reliability of measurements across various applications, making traceability a cornerstone of modern scientific, industrial, and commercial activities.

An uncertainty budget is necessary because it combines all potential sources of error and their contributions to the overall measurement uncertainty. This detailed analysis enables better decision-making and confidence in the results, ensuring that measurements are reliable and fit for their intended purpose. Furthermore, it supports compliance with quality standards and regulatory requirements, essential in industries where precision and safety are paramount. Clarifying the distinctions between precision, reproducibility, and uncertainty is a focal point, as these are critical in interpreting the quality of quantitative data. Metrological principles in chemistry define measurement quality not only by precision or closeness to a true value but by the validity of results as confirmed through uncertainty estimates.

The course is designed for beginners in the field as well as for those seeking to update and reinforce their foundational knowledge.

(A-3)

Multimodal imaging: Sample preparation, instrumentation and data evaluation

Uwe Karst, Katharina Kronenberg, Christine Verlemann, Peter Niehaus and Michael Sperling

Institute of Inorganic and Analytical Chemistry, University of Münster, Corrensstr. 48, 48149 Münster, Germany
E-mail: uk@uni-muenster.de

To study the distribution and the biological effects of metallopharmaceuticals and other metal species in the human, animal or plant organism, imaging methods at the tissue scale with lateral resolution in the lower micrometer range have been found to be particularly valuable.

Traditionally, pathologists use formalin fixation and subsequent paraffin embedding as standard method for sample preparation prior to optical microscopic investigation of the tissue samples. (Immuno)histological staining may provide additional valuable information on the samples. However, rinsing steps during these procedures may either remove analytes or add contaminations. Whereas this is not an issue for optical microscopy, chemical imaging techniques often require the use of fresh frozen tissue samples instead. In this short course, possibilities and limitations of sample preparation techniques for chemical imaging will be presented and critically discussed under consideration of different chemical species.

While a single imaging method can provide valuable chemical data on tissue samples, the complementary use of a multitude of imaging methods may allow to obtain essential information on complex samples. These techniques include, among others, optical microscopy (transmission, autofluorescence etc.), micro-X-ray fluorescence spectroscopy (µXRF) for elemental pre-screening, laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) for elemental quantification, quantum cascade laser (QCL)-based infrared (IR) microscopy for tissue identification and matrix-assisted laser desorption/ionization-mass spectrometry (MALDI-MS) for the analysis of molecular distributions. The respective techniques will be introduced and discussed with respect to the selection of appropriate sample carriers. Strategies to gain maximum information from the complementary methods and to commonly evaluate multimodal data will also be discussed.

(A-4)

Hyphenated techniques for elemental speciation analysis in environmental samples – an overview

Daniel Pröfrock

Helmholtz-Zentrum Hereon, Institute of Coastal Environmental Chemistry, Department Inorganic Environmental Chemistry, Max-Planck-Str. 1, 21502 Geesthacht, Germany

E-mail: Daniel.Proefrock@hereon.de

The importance of speciation analysis has been predicted already more than 30 years ago. In parallel the outstanding progress made within this field strongly contributed to the improved understanding of the functional significance and possible interactions of trace elements with ecological compartments and biological systems.

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

Various new strategies and analytical tools for the complementary application of elemental mass spectrometry (e.g. inductively coupled plasma mass spectrometry (ICP-MS)) which is still generally known as only a “metal” detector beside techniques such as ESI-MS and MALDI-TOF MS and their utilization for new, “non-traditional” application areas have been developed over the years.

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

(A-5)

Electrical plasmas for molecular mass spectrometry

Jacob T. Shelley

Department of Chemistry and Chemical Biology, Rensselaer Polytechnic Institute, Troy, NY 12180 USA

Electrical plasmas and mass spectrometry (MS) have gone hand-in-hand since J.J. Thomson used a reduced-pressure glow discharge as the first ionization approach in the discovery/development of mass spectrometry. Historically, plasma ion sources were used for these experiments because they were one of the few known sources of gas-phase ions at the time and they were relatively simple to setup and operate. However, developments in plasma ionization have continued to inform and motivate advances in other areas of MS long after the work of Thomson and Aston. More recently, atmospheric-pressure (AP) plasmas have been explored for a variety of purposes including direct mass-spectrometric analyses, molecular and elemental ionization, surface modification, chemical synthesis, and environmental remediation.

Atmospheric-pressure discharges are unique in that they simultaneously produce highly energetic species as well as lower energy chemical reagents. Furthermore, it has been shown that plasma conditions (e.g., power, gas composition, etc.) can be slightly altered to favor one condition, even on the timescale of analysis. This short course will cover emerging trends in AP plasma source usage in chemical and analytical sciences, with an emphasis on methods that utilize MS.

(B-1)

Laser Induced XUV Spectroscopy (LIXS): the future of LIBS is now

Davide Bleiner[1,2]

[1]University of Zurich – Dept. Chemistry, University of Zurich, Winterthurerstrasse 190, CH-8057 Zurich, Switzerland
[2]Empa – Swiss Federal Laboratories for Materials & Technology, Überlandstrasse 129, CH-8600 Switzerland

E-mail: davide.bleiner@uzh.ch

Laser-induced breakdown spectroscopy (LIBS) is a rapid in-situ microanalytical technique for elemental mapping. A laser pulse generates a plasma on a sample surface, whose spectral signature is assumed to be indicative of the parent material chemistry on the spot. Affected by poor precision, due to plasma flicker noise, LIBS is limited in delivering a huge potential for surface heterogeneity spotting. A better precision means the capability to spot trace heterogeneities in materials.

Laser-induced XUV spectroscopy (LIXS) [1-3], works similarly to LIBS, while it collects the early stage plasma emission in the extreme ultraviolet and soft X-rays. XUV emission is associated with the hot and dense point plasma, at the onset of the expansion. Specific electron-ion processes in the LIXS plasma are discussed, showing why LIXS improves the limits of detection and the precision of LIBS.

Enabling for LIXS is the realization of a specific XUV spectrometer using variable line spacing gratings. Details on such instrumental challenges and solutions are discussed in detail. A selection of applications in the analysis of materials is discussed, showing that LIXS' strength is the ability to determine light elements as well as halogens with high signal-to-noise ratio and fractionation-free response. Examples and exercises are proposed to make the participant exploring themselves.

[1] Bleiner, Davide, et al. "Laser-induced XUV spectroscopy (LIXS): From fundamentals to application for high-precision LIBS." *Spectrochimica Acta Part B: Atomic Spectroscopy* 204 (2023): 106668.
[2] Qu, Di, et al. "High-precision mapping of fluorine and lithium in energy materials by means of laser-induced XUV spectroscopy (LIXS)." *Spectrochimica Acta Part B: Atomic Spectroscopy* 181 (2021): 106214.
[3] Qu, Di, and Davide Bleiner. "High-Precision Micro/Macro-Analysis with Laser-induced XUV Spectroscopy (LIXS): Highlights of Analytical Sciences in Switzerland." *CHIMIA* 76.1-2 (2022): 153-153.

(B-2)

How to reduce ICP-OES interferences for 69 elements

Thomas J. Kozikowski and Mike L. Booth

Inorganic Ventures, Christiansburg, VA 24136 USA

E-mail: tkozikowski@inorganicventures.com

Inorganic Ventures is a certified reference material manufacturer which specializes in providing high quality custom solutions for ICP users worldwide. Although custom solutions are certified gravimetrically by balance, there is still a need to verify that the solutions manufactured at the bench are free from gross human error or unexpected chemical incompatibility. Our Quality Control group was using a method developed over 20 years ago for axial view ICP-OES and was fairly limited for calibration range (0 to 1ppm) due to the large number of elements of interest. A new method was desired with the main goal of increasing throughput by cutting down on sample preps required for a custom solution to fit varying concentration analytes on the narrow calibration curve. With the use of radial view, the expansion of the calibration range was desired to achieve this goal. However, higher concentration standards bring a host of chemical compatibility concerns along with spectral interferences.

This short course will go through the method development steps required to arrive at a single method capable of routinely checking 69 elements on ICP-OES while accounting for chemical stability as well as spectral interferences without the use of an internal standard. We will take a deep dive into the major chemical and acid matrix compatibility issues that occur when designing calibration standards. We will also navigate through specific spectral interferences worth noting and show how splitting up calibrations curves within the same run can allow for the screening of complicated custom solutions with little difficulty. A custom-built interference correction table will also be shared to illustrate its effectiveness when minor interferences cannot be avoided. Finally, we will touch on the validation steps required to ensure that subsequent runs on this method are valid and still fit for purpose. This will include long term stability of the calibration standards, calibration verification, matrix matching concerns, and finally instrument setup.

(B-3)

SIMS and related techniques for elemental and isotopic imaging at nanometer scale

Dirk Schaumlöffel

CNRS/Université de Pau et des Pays de l'Adour, E2S UPPA, IPREM UMR 5254, Hélioparc, 2, avenue du président Angot, F-64053 Pau, France

E-mail: dirk.schaumloeffel@univ-pau.fr

In this short course, element specific imaging techniques with a focus on secondary ion mass spectrometry (SIMS) will be presented. The principles of SIMS including ion sources and the ionization process will be covered as well as the difference between dynamic and static SIMS techniques. This course discusses also briefly related X-ray based techniques such as electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS, TEM-EDS). In a second part nanoscale secondary ion mass spectrometry (NanoSIMS) will be the main subject for chemical and isotopic imaging at the nanometer scale: principle, ion sources, ion transmission, lateral resolution, mass resolution, and useful yield. This course includes furthermore challenges for sample preparation especially for biological material as well as solutions for correlative imaging with related techniques. Finally, challenges and limitations of image date treatment will be discussed and application examples from our laboratory will be presented.

(B-4)

Complementarity of ICP-MS and electrospray MS^n in advanced speciation analysis

Joanna Szpunar

Institute of Analytical Sciences and Physicochemistry of Environment (IPREM) UMR 5254, National Research Council of France (CNRS), Hélioparc, 64 053 Pau, France

E-mail: joanna.szpunar@univ-pau.fr

The short course will present the basics of “soft" ionization ESI-MS^n and evaluate its role in the detection and identification of metal/metalloid-containing biomolecules in comparison with classical ICP-MS ("hard" ionization) based methods.

The idea of the parallel elemental and molecular mass spectrometric detection in HPLC-based speciation techniques has been explored for many years. It has allowed standardless identification of, unknown so far, species appearing in the HPLC-ICP-MS chromatograms as the results of growing sensitivity of plasma MS.

However, analytical approaches to be used depend on the molecular weight and he stability of the species of interest. The procedures suitable for studies of low molecular weight (LMW) covalent and coordination complexes as well as for heteroatom-containing proteins will be presented with a focus on the crucial experimental steps bearing risk of species loss, transformation and/or misidentification.

The perspectives for ESI-MS^n to partially replace ICP-MS in speciation analysis, resulting from the progress in the development of high-resolution high accuracy molecular FT-MS, will be highlighted. Nowadays, the coupling of HPLC-ESI Orbitrap MS allows the detection of the heteroatom-isotopic pattern (an equivalent of the element-specific signal) in mass spectra with the low- and sub-ppm mass accuracy. A cascade of product ion mass spectra with the preservation of the isotopic pattern can be produced at the sub-ppm mass accuracy largely facilitating the analyte structure elucidation. Thus, in many speciation-related applications in the analysis of biological samples, the ESI MS has been not only complementing ICP-MS, but also slowly gaining a place of standalone technique.

The course will be illustrated by the findings of several projects focused on identification and quantification of species formed as a result of in-vivo interactions of metals with biomolecules.

(B-5)

LA-ICP-MS mapping; from fundamentals to the best image quality maps

Martin Šala

Department of Analytical Chemistry, National Institute of Chemistry, Hajdrihova 19, Ljubljana SI-1000, Slovenia

The short course will cover the basics of LA-ICP-MS mapping and provide insights into optimizing parameters to achieve the highest image quality possible. However, before adjusting your parameters, it's essential to understand the equipment currently used, its advantages, drawbacks, and how to leverage it effectively.

In addition to typical experimental parameters like laser fluence, beam size, and repetition rate, we'll delve into calibration possibilities and various approaches. By understanding their interplay, you'll be equipped to plan and execute your experiments confidently, leading to excellent quantified elemental maps.

(C-1)

Sample classification based on elemental fingerprinting: Introduction and practical guideline

Andreas Limbeck and Lukas Brunnbauer

TU Wien, Institute of Chemical Technologies and Analytics, Getreidemarkt 9/164-I²AC,
1060Vienna, Austria

E-mail: andreas.limbeck@tuwien.ac.at; Lukas.brunnbauer@tuwien.ac.at

In various fields of modern science but also for routine applications in industry, food inspection, and health care, there is an increasing need to discriminate samples with differences in behavior or origin. At this point Analytical Chemistry plays a fundamental role since analysts have to be responsible for developing methodologies that allow the confirmation of provenance or authenticity, the detection of product adulteration and verifying their compliance with labeling.
In recent years, much progress has been made in sample classification based on elemental fingerprinting techniques. To achieve this, elemental analysis is combined with multivariate statistical analysis of the obtained data to gain information about differences or variations within the investigated samples.

In this short course, we will briefly present the concepts and approaches used for assessing elemental contents and provide an overview of common chemometric methods and their potential for sample classification. Finally, we will discuss some descriptive application examples from the fields of food analysis, geology, life sciences and material science.

(C-2)

Engineered nanomaterials: Reference methods, reference materials and documentary standards in support of current regulations

D. Bartczak and H. Goenaga-Infante

LGC National Measurement Laboratory, Queens Road, Teddington, UK
E-mail: dorota.bartczak@lgcgroup.com

Nanotechnology impacts most areas of our lives with thousands of nanomaterial (NM) based commercial products, ranging from food, personal care and healthcare to paints, inks, electronics and automotive, currently on the market[1]. Manufacturers of such NM based products are required to comply with the emerging regulation, for example with the EU Directives (1223/2009, 2017/745, 2015/2283, 1935/2004), which specify the physicochemical characteristics of NMs permitted for commercial use.

For this reason, recent research efforts have been largely focused on the development, validation and harmonisation of reference methods for the characterisation and quantification of NM in regulatory context, as well as on the development and commercialisation of quality control and reference materials needed for instrument calibration and day-to-day method’s performance verification.

This short course will provide the participants with an overview of the currently available quality control and reference materials, which to date are still very limited, especially materials characterised for particle number concentration. In this context, examples of currently available reference methods for quantification of NM in terms of mass as well as number-based concentration will be given. Special attention will be paid to methods offering direct traceability to the SI unit of kg and to methods which are based on ICP-MS. Performance of other popular laboratory-based approaches in comparison with reference methods, verified under international laboratory comparison will be discussed. Overview of relevant standardisation activities undertaken under ISO and CEN committees will also be provided as part of this course. The course will provide participants with opportunities to ask questions and participate in round table discussions, but no practical exercise is planned as part of it.

(C-3)

Short course on fluorine detection with ICP-MS and ESI-MS and applications

Joerg Feldmann and Viktoria Müller

TESLA – Analytical Chemistry, University of Graz, 8010 Graz, Austria

E.mail: joerg.feldmann@uni-graz.at

Fluorine is one of the elements which cannot directly be detected by conventional ICP-MS. This course will give some background, how environmental analysis for organofluorines such as F-containing pesticides and F- containing pharmaceuticals and PFAS (Per and polyfluorinated alkylated substances) is routinely analysed by target LC-MS/MS. Furthermore, will be demonstrated how ICP-MS can be used for non-target HR-ESI-MS. Applications about PFAS in food contact materials, ski wax, accumulation in wildlife as well as fluorinated compounds in sewage water. Additionally, we will feature how LA-ICP-MS and sp-ICP-MS can be conducted for fluorine.

(C-4)

Arsenic speciation analysis with ICP-MS as element-selective detector

Walter Gössler

University of Graz, Austria

Nowadays it is well accepted that speciation analysis is important to estimate a risk originating from our food. Especially arsenic is often studied in the scientific literature. In our food arsenic may be present either in toxic for or in a nontoxic form. The inorganic and trivalent forms of arsenic exhibit a significantly higher toxicity than the organic forms. In the marine ecosystem the situation is rather simple as marine animals mainly contain arsenobetaine and marine algae contain predominantly arsenosugars. Arsenobetaine is of no known toxicity. Arsenosugars are converted in the human body to smaller arsenicals such as dimethylarsinic acid which is far less toxic than inorganic arsenic. In the terrestrial ecosystem the situation is more complex because samples are more divers in their arsenic speciation. Arsenic might be present in its inorganic form or other less toxic forms of arsenic are dominating in the samples. Therefore, it is important to determine the chemical form arsenic when the total arsenic concentration exceeds the legal limit.

For arsenic speciation analysis of solid samples, the arsenic species are often extracted with mild extractants such as methanol/water mixtures or weak acids such as phosphoric acid, acetic acid or even dilute nitric acid. After the extraction has been optimized for a certain analyte and sample matrix and a sufficiently high extraction yield has been obtained the arsenic compounds have to be determined in this extract. Therefore, liquid chromatography employing anion-exchange and/or cation-exchange stationary phases are used to separate the arsenic compounds. Due to the fact that the legal limits are quite low ICPMS is commonly used as an element-selective detector.

In the course various aspects of the analytical process during arsenic speciation analysis are discussed. This covers pitfalls that can occur during the total arsenic determination as well as problems that might occur during the separation of the arsenic compounds. The carbon enhancement effect for arsenic during total arsenic determinations and during chromatographic separation will be shown on various examples and solutions for these problems are offered.

Commonly used separation techniques used for arsenic speciation analysis are introduced and discussed.

(C-5)

Isotopic analysis using ICP-MS: Fundamentals and applications

Lana Abou-Zeid and Frank Vanhaecke

Ghent University, Department of Chemistry, Atomic & Mass Spectrometry − A&MS research group, Ghent, Belgium

Elements with two or more isotopes often show natural variation in their isotopic composition owing to mass-dependent fractionation. This occurs because of different isotopes engaging in physicochemical and biogeochemical reactions in a slightly different manner. In contrast to thermal ionization mass spectrometry (TIMS), the gold standard for isotope ratio measurements, inductively coupled plasma-mass spectrometry (ICP-MS) showed to be an attractive tool in this context mainly due to its powerful ICP source that allows efficient ionization of elements with high ionization energy (>7.5 eV), as well as its simple and highthroughput introduction system[1]. As standard ICP-MS was mainly designed for sensitive elemental analysis and not specifically for isotopic analysis, a modest precision ≥0.05% RSD can be obtained. This precision is sufficient for quantifying induced variations in isotopic composition in the context of tracer experiments and for revealing relatively large natural variations in the isotopic compositions of specific isotopic systems as for example lead (Pb), for tracing Pb pollution sources in the environment. Multi-collector inductively coupled plasma-mass spectrometry (MC-ICP-MS) has been specifically designed for high-precision isotope ratio measurements (down to 0.001% RSD measurement precision) as it offers simultaneous detection of all isotopes of the element of interest, enabling very small changes in isotopic composition to be revealed and quantified.

Identifying natural isotope ratio variations showed be an important source of information to gain deeper insights into physicochemical processes. in many fields such as geochemistry, cosmochemistry, the environmental sciences and more recently, also in biomedical contexts[2]. While precision is substantially improved with MC-ICP-MS, accuracy can be compromised using both single and multi-collector ICP-MS by (i) the occurrence spectral interferences which causes overlap with the signal of one or more isotopes of the element of interest and (ii) instrumental mass bias which results from the preferential transport of lighter isotopes from the ion source to the detector of the mass spectrometer[3].

Overcoming spectral interferences and correcting for instrumental mass bias can be addressed in several ways as will be presented during this course. In addition, the capabilities and limitations of single- and multi-collector ICP-MS instrumentation for isotope ratio measurements will be discussed. Finally, the capabilities of isotopic analysis across various fields will be illustrated using application examples from the authors’ lab or reported in the literature.

1 F. Vanhaecke, L. Balcaen and D. Malinovsky, *J. Anal. At. Spectrom.*, 2009, **24**, 863.

2 M. Costas-Rodriguez, J. Delanghe and F. Vanhaecke, *TrAC Trends in Analytical Chemistry*, 2016, **76**, 182−193.

3 P. A. Penanes, A. R. Galan, G. Huelga-Suarez, J. A. Rodriguez-Castrillon, M. Moldovan and J. I. Garcia Alonso, *J. Anal. At. Spectrom.*, 2022, 37, 701−726.

(D-1)

ICP-MS based analytical strategies to track nanoparticles in biological systems

Maria Montes-Bayón

Department of Physical and Analytical Chemistry. Faculty of Chemistry. University of Oviedo.
C/ Julián Clavería 8, 33006, Oviedo, Spain.
Health Research Institute of the Principality of Asturias (ISPA). Avda. Hospital Universitario s/n, 33011, Oviedo, Spain

The use of nanoparticles in the industry has been increased in the last decade for many applications like catalysis, additives in textiles and polymers, as antibacterial agents, in medical applications like drug and gene delivery, in bioimaging, etc. Nevertheless, the impact of these nanomaterials on the environment and on the biological systems is still not well defined. The research about their potential toxicity to living organisms is gaining interest in the last years. Reliable analytical methods are therefore urgently required for a better in deep characterization of these new materials. ICP-MS based analytical methods offer different possibilities to obtain information related to the targeted particle itself as well as their possible effects on living biological systems. In particular, single particle ICP-MS strategies and the hyphenation of mass spectrometry with *state of the art* separation techniques further provides valuable information related to size, shape and state of agglomeration of the nanoparticles present in the investigated samples.

During the short course the different sample preparation strategies to obtain stable nanoparticle suspensions will be highlighted. Also, the use of SP-ICP-MS and HPLC-ICP-MS analysis methods will be illustrated by focusing on their use for the evaluation of the presence of nanoparticles in biological samples: yeast, cells and tissues.

(D-2)

Nanomaterials characterization by ICP-MS in single particle mode and related techniques

Carsten Engelhard[1,2]

[1]BAM, Bundesanstalt für Materialforschung und -prüfung, Richard-Willstätter-Straße 11, 12489 Berlin
[2]University of Siegen, Department of Chemistry and Biology, Adolf-Reichwein-Str. 2, D-57076 Siegen, Germany
E-mail: carsten.engelhard@bam.de

Single particle inductively coupled plasma mass spectrometry (spICP-MS) is a powerful technique that can provide unique information on the elemental composition of nanoparticles (NPs). The method allows determining the size, size distribution, and particle number concentrations of NPs in suspensions after a mere few minutes of measurement. In addition, spICP-MS has also proven to be useful to detect nanoparticles at environmentally relevant concentrations.

In this short course, fundamental principles of spICP-MS, instrumentation, and selected applications will be discussed. This course will begin with a tutorial on spICP-MS basics including sampling, sample preparation, microsecond time-resolved detection, data processing, and data evaluation. A particular emphasis will be placed on measurement artifacts that can arise during data acquisition, which – if unnoticed or uncorrected – may lead to errors in results and particle size histograms. In the second part, selected applications will be highlighted including the detection of NPs in complex matrices and environmental samples.

This course is intended for those with a background in ICP-MS but beginners are also very welcome to attend.

(D-3)

Practical approaches to use reaction gases in ICP-MS triple quadrupole

Karl Andreas Jensen

Centre for Environmental Radioactivity (CERAD CoE), Faculty of Environmental Sciences and Natural Resource Management (MINA), Norwegian University of Life Sciences (NMBU), NO-1432 Ås, Norway

e-mail:karl.jensen@nmbu.no

This short course will show how to proceed in method development working on an ICP-MS triple quadrupole. The course is aimed at users of ICP-QQQ who would like to get more knowledge about the reactions in the reaction cell and how to change these reactions. The following questions will be addressed:

- Does the interference or the analyte have an exothermic or endothermic reaction with the added gas?

- How does the collision energy affect the reactions in the cell?
- Why is the ionization energy important?

- Which gases are suitable for the different analytes and mass interferences? What are the products?

- Regarding manually optimizing of the cell parameters, which parameters are important? How do these affect the reaction efficiency?

These questions will be answered with examples on polyatomic-, doubly charged-, and isobar mass interferences.

(D-4)

Make your lecture count!

Gary M. Hieftje

Department of Chemistry, 800 East Kirkwood Ave., Bloomington IN 47405 USA

Home address: 980 Cape Marco Drive, Unit 1205, Marco Island, FL 34145 USA

e-mail: Hieftje@indiana.edu

In “The Making of a Scientist” (1953), Anne Roe said, “Nothing in science has any value to society if it is not communicated…”. Among the ways by which this communication is achieved, the scientific lecture is one of the most important. Yet, too many of us squander opportunities to communicate in this way by crafting and delivering lectures that are not as effective as they might be.

In this short course, based on a long-running series of seminars at Indiana University, suggestions will be offered on how to prepare for, present, and benefit from scientific lectures, be they in a classroom environment, technical conference, local seminar, job interview, or short course.

14 Poster Abstracts

(P-1)

Speciation analysis of Cr(III) and Cr(VI) using µLC-ICP-MS

Jelle Verdonck[1], Katrien Poels[1], Jeroen Vanoirbeek[1], Radu Corneliu Duca[1,2], Lode Godderis[1,3] and Erk Smolders[4]

[1] Centre for Environment and Health, Department of Public Health and Primary Care, KU Leuven (University of Leuven), Leuven, Belgium
[2] Environmental Department Health Protection, Laboratoire National de Santé (LNS), Dudelange, Luxembourg
[3] IDEWE, External Service for Prevention and Protection at Work, Heverlee, Belgium
[4] Division of Soil and Water Management, Department of Earth and Environmental Sciences, KU Leuven (University of Leuven), Leuven, Belgium

Chromium (Cr) is a transition element that exists in oxidation states ranging from - 2 to +6. The common stable ones in the environment are trivalent Cr(III) and hexavalent Cr(VI) chromium. Cr(III) is an important micronutrient for the human body, while Cr(VI) is highly toxic and carcinogenic. The environmental concentrations of both oxidation states are low. Due to the differences in toxicity between Cr(VI) and Cr(III) compounds, speciation of Cr is very important. Therefore, an improved sensitive and robust method for the simultaneous determination of Cr(III) and Cr(VI) in water samples (saliva, waste water and nutrional solution) has been developed.

The method uses a hyphenated micro liquid chromatography (µLC) system coupled to inductively coupled plasma mass spectrometry (ICP-MS). The optimised method incorporates a pH adjusted EDTA complexation step to stabilise Cr(VI) and Cr(III). The µLC system uses an anion exchange micro-sized column to separate the Cr species. Cr(III) and Cr(VI) were separated with different retention times at 170 and 230 sec, respectively. The method was optimized and validated by spiking Cr(III) and Cr(VI) in various water samples. Furthermore, the method was validated using a drinking water proficiency testing material sample. The developed method can be used for rapid routine determination of chromium species with high precision and reliability.

(P-2)

The presence and behavior of titanium and silver nanoparticles in a water treatment plant and wastewater treatment plant located in the Llobregat River (Barcelona, Spain)

A. Díaz[1], E. Guiot[1], I. Perez[1], E. Peña-Vázquez[2], M.C. Barciela-Alonso[2]. M. Aboal-Somoza[2] and P. Bermejo-Barrera[2]

[1] *Aigües de Barcelona (Agbar). Laboratory. General Batet 5-7, 08028 Barcelona, Spain*
[2] *Trace Element, Spectroscopy and Speciation Group (GETEE), Instituto de Materiais (iMATUS), Faculty of Chemistry, University of Santiago de Compostela, Av. das Ciencias, s/n 15782, Santiago de Compostela*

Recently, some European Health Agencies are following up the presence and behavior of some specific metallic particulates, such as silver and titanium nanoparticles that can be found in the environment from the manufacturing of different industrial applications, such as food or cosmetic additives.
In this work we present a study of these nanoparticles and their removal efficiency in a Drinking Water Treatment Plant (DWTP) and a Wastewater Treatment Plant (WWTP), both of them located in Llobregat River (Barcelona, Spain).

The methodology used for the determination of silver and titanium nanoparticles was *Cloud Point Extraction* (CPE). The extract is direct analyzed by ICP-MS and single-particle (SP-ICP-MS) using a specific software (Perkin Elmer NexIon 2000) to assess their concentration and size distribution.
The procedure used is presented in Figure 1 and has been developed by Bermejo's team in Santiago de Compostela.

92% of silver found in raw wastewater was in nanoparticle form and the more frequent size detected were 16.4 nm.
Less than *40% of titanium* was in nanoparticle form with a more frequent *size of 86 nm for raw wastewater and 56.5 nm for Llobregat river. There were no detection by SP-ICP-MS in outlet sample for both facilities.*

According to the results obtained, the *removal efficiency in both, drinking and wastewater treatment plants were about 90%* showing a good feasibility of both facilities to eliminate these types of contaminants.

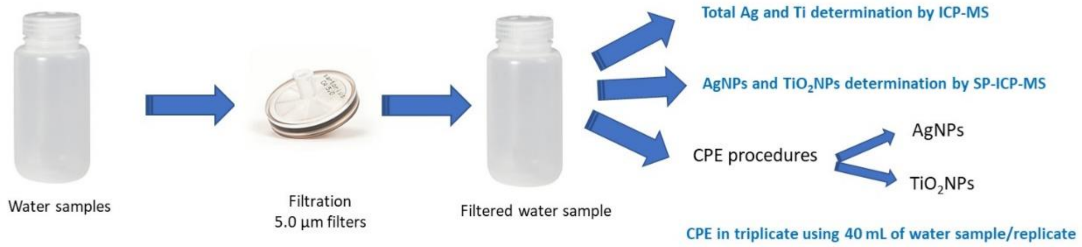


Figure 1. Protocol used for the analysis of samples.

(P-3)

Improving image quality and pixel resolution via laser pulse dosage increase for fast imaging of large areas with LIBS

Jakob Willner, Lukas Brunnbauer, David Gibbs, Maximilian Podsednik, and Andreas Limbeck[1]

TU Wien, Institute of Chemical Technologies and Analytics, Getreidemarkt 9/164-IAC, 1060 Vienna, Austria

Laser induced breakdown spectroscopy is a powerful method for spatially resolved and fast elemental analysis of solid samples and represents a handy tool for elemental mapping of micro-structured samples with relatively high sensitivity. However, besides the pulse energy of the laser, the sensitivity of the analysis is further depending on the spot size of the laser pulse and, thus, is competing with the lateral resolution. By applying larger spot sizes and an increased laser dosage, which is the measurement with partially overlapping shots, in imaging experiments followed by the according post data treatment the pixel resolution can be increased while maintaining higher signal-to-noise-ratios compared to the measurement directly with smaller spot sizes.

This work explores the influence of different spot sizes and laser dosages on the image quality regarding pixel resolution and sensitivity using a 193 nm excimer laser and a multichannel broadband LIBS spectrometer for imaging experiments on structures of printed circuit boards and microchips. While main and minor constituents, such as copper, tin, zinc, lead, can be measured simultaneously with broadband-LIBS, an additional high-resolution spectrometer with ICCD camera gives access to selected traces, e.g., impurities and contaminants inside materials utilized in PCB and microchip manufacturing.

(P-4)

Thin water films transport compounds from the leaf surface into the plant tissue - Laser ablation ICP-MS reveals the pathways

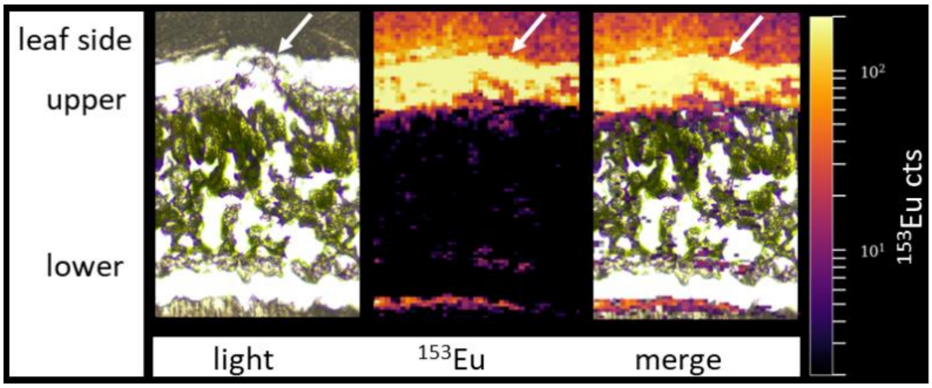
Max Frank[1], Francesca Siracusa[2], Daniel P. Persson[1], Rajmund Mokso[2], Søren Husted[1]

[1] Department of Plant and Environmental Sciences , University of Copenhagen, Denmark,
[2] Department of Physics, Technical University of Denmark, Denmark.
E-mail: maf@plen.ku.dk

Conventional fertilizers are often inefficient and contribute to major environmental problems, ranging from biodiversity loss to climate change. Applying fertilizers directly to the leaves instead of soils has the potential to significantly increase the uptake efficiency into crop plants. Foliar uptake of solutes and colloids varies greatly between different plant species, and the uptake pathways into plant tissues are largely unknown [1]. Current evidence points to stomata as the major entry points for aqueous solutions. For the uptake to take place, a thin layer of water needs to wet a hydrophobic stomatal cell surface, a process called hydraulic activation of stomata (HAS). Solutes and colloids are believed to travel along these thin water films into the cavity below stomata. In this study, we combine a laser ablation ICP-MS (LA-ICP-MS) with confocal laser scanning microscopy (CLSM) and high resolution x-ray tomography (µCT) to investigate ion and particle uptake into leaves of barley and potato plants.

CLSM gives a good overview about the effect of different topographies on the distribution of solutions on varying leaf surfaces. However, the lipophilic properties of many fluorescent dyes are suboptimal to follow thin water films on a waxy leaf surface, and the method is limited by the laser penetration depth, refractive properties of leaf tissues, and plant tissue autofluorescence. We overcome this by using µCT, where contrast agents introduce density variations and phase differences to create contrast. While this gives more realistic insight into the penetration of leaves by water, artefacts are introduced easily, which makes image interpretation often difficult. It gives no information about the chemical composition of the volume investigated. Elemental mapping of thin leaf sections generated with LA-ICP-MS is the only method that allows us to verify that the contrast is generated by the solution that was applied to the leaf, and it reveals how nano particles actually distribute and dissolve within the tissue. A gel based calibration curve enables us to semi-quantitatively estimate the uptake efficiency into the leaf tissue. LA-ICP-MS also helps to investigate the varying distribution patterns of ions or colloids with different properties within the leaf. As such, LA-ICP-MS is the core method when it comes to verification of data from CLSM and µCT, and it plays a key role in investigating foliar uptake pathways into leaf tissue.

Figure: Thin section of a potato leaf. LA-ICP-MS allows to investigate how stomata (arrows) are penetrated by water transporting nano particles spiked with Eu, here at a 5µm pixel size.



[1] Husted, S. et al. What is missing to advance foliar fertilization using nanotechnology? *Trends in Plant Science* (2023), 28(1), 90–105.

(P-5)

Rapid microwave-assisted acid digestion of heavy petroleum products for trace metal analysis by ICP-OES and ICP-MS

Markus Michaelis[1] and Glenn Müller[2],

[1] Anton Paar GmbH, Graz, Austria
[2] Dipl.ing. Houm AS, Oslo, Norway

Determining metals in petroleum products is increasingly important. While established methods[1] work for simple products, heavy mineral oils (marine fuels, crude oils, etc.) require time-consuming procedures involving burning, charring, and fluxing, taking up to 2 days.

This study presents microwave-assisted acid digestion as a faster and safer alternative, reducing sample preparation time to 2.5-3 hours. Analytical data from digesting ASTM proficiency testing samples and NIST SRM will be shown. Two vessel systems will be compared: a closed system with high-speed pressure monitoring and a SmartVent system for controlled pressure release.

This method offers comparable or improved analytical performance while significantly reducing energy consumption, reagent use, waste generation, and operator time.
This novel approach provides a faster, safer, and more efficient method for analyzing metals in complex petroleum products.

[1] IP 501/05, Energy Institute, London, (2005)

(P-6)

Li-ion battery materials digested for ICP-OES analysis

Markus Michaelis[1], Gisela Fauler[1] and Glenn Müller[2]

[1] *Anton Paar GmbH, Graz, Austria*
[2] *Dipl.Ing. Houm AS, Oslo, Norway*

The performance of battery materials is related to their composition and the decomposition products formed over their lifetime. The chemical composition of all battery components is crucial to better understand aging effects and extend battery service life. Therefore, reliable element and trace element analysis is a must to determine stoichiometries of the active materials, their changes along charge/discharge cycles, and impurities.

Since the chemical compositions of battery materials differ a lot, appropriate sample preparation for further (trace) element determination is a crucial and often an error-prone step in the whole analytical process.

This study demonstrates how the microwave-assisted acid digestion facilitates subsequent ICP-OES or ICP-MS analysis of all types of battery materials, ranging from lithiated metal oxides or phosphates (cathodes), graphite-based material (anodes) to polymer foils (used as separator) and black mass, the powdered mix of complete end-of-life batteries.

Two different microwave digestion systems will be on display, digesting a variety of real-life samples together in one run and showing how convenient and efficient sample preparation can be for further element. Analytical results from the subsequent ICP-OES analysis will also be shown to complete the presentation.

(P-7)

Ever lower limits challenge ICP-MS food analysis: How microwave acid digestion helps

Klaus Pichler-Jöbstl and Markus Michaelis,

Anton Paar GmbH, Graz, Austria

Stricter regulations for food safety like the legislative proposal of the U.S. Baby Food Safety Act of 2021, with lower limits for contaminants like arsenic (As), cadmium (Cd), lead (Pb), and mercury (Hg) in baby food, necessitate highly sensitive analytical techniques. Sample preparation, particularly mirowave closed-vessel acid digestion, plays a crucial role in achieving accurate results.

This study evaluates a robust and efficient nitric acid digestion method using Multiwave 7000 for analyzing trace elements in infant and toddler food. The method prioritizes maximum sample weight, minimal acid use, complete digestion, and single-run processing for various food compositions.

Eight food samples with varying water, carbohydrate, fat, and protein content, along with reference material NIST 1570a, were digested and analyzed by ICP-MS based on FDA method 4.7. Clean quartz vials and ultrapure reagents ensured accurate results.

- Arsenic (As) enhancement: 200 µL isopropanol was added post-digestion to mitigate carbon interference and improve As precision.

- Mercury (Hg) stabilization**:** 0.2 mL HCl was added post-digestion to stabilize Hg in solution.

Two samples, a dry corn wafer and a wet fruit puree, were spiked with target elements at concentrations proposed in the Baby Food Safety Act. All recoveries for As, Cd, Pb, and Hg fell within the 80-120% range mandated by FDA method EAM 4.7, with relative standard deviations below 7%.

The optimized digestion method offers a reliable and efficient approach for analyzing trace elements in diverse food samples, enabling laboratories to meet stringent food safety regulations.

(P-8)

Acid digestion for PGM recycling analysis with ICP-MS

Markus Michaelis and Peter Kettisch

Anton Paar GmbH, Graz, Austria

Platinum group metals (PGMs) are valuable materials for various industries, their analysis for main metal content and impurities is of high importance in producing the metals and in the recycling process. The preferred method is acid digestion and subsequent measurement with ICP-MS or ICP-OES.

The digestion of PGMs, however, is challenging, since it requires high temperatures, long reaction times and highly “aggressive” acids like aqua regia and perchloric acid. While many microwave digestion systems cannot operate under these conditions, it will be shown how the concept of a *Pressurized Digestion Cavity* (PDC) provides these extreme reaction conditions required for the complete dissolution of the most resistant noble metals.

Powders of platinum, palladium and rhodium were selected in order to demonstrate the digestion of pure PGMs. Furthermore, a palladium catalyst and a Si-Mg-Fe ore were digested. The latter one is a laboratory-internal standard and contains known trace amounts of PGMs as a reference for the evaluation of results.

Platinum and palladium can be digested with aqua regia, rhodium requires the addition of perchloric acid to the hydrochloric acid for complete digestion. Furthermore, the importance of stirring during the reaction was investigated.

For the zeolite-based palladium catalyst the reagent mixture was optimized for a complete digestion of the silicate carrier. However, for catalysts only the PGM content counts, so leaching is normally sufficient.

(P-9)

Acid digestion of high purity PGMs including rhodium, iridium, and ruthenium

Markus Michaelis and Peter Kettisch

Anton Paar GmbH, Graz, Austria

Digestion of Platinum group metals (PGMs) like palladium, rhodium, iridium, and ruthenium for impurity analysis is quite challenging. It requires high temperatures, long reaction times and highly aggressive reagents, like aqua regia, pure concentrated hydrochloric acid or even a mixture with perchloric acid. The task becomes more challenging when the analysis for Silica is required on trace levels.

Sealed quartz vessels in the autoclave-type microwave heated digester “Multiwave 7301” allow for safe usage of these reagents, but can compromise the accurate determination of Si.
We present a safe and reproducible digestion technique which helps to overcome this limitation: for Si determination the digestion of up to 100 mg of PGM powder is performed in PTFE-TFM insert vials within the sealed quartz vessels. For the complete dissolution of rhodium, iridium, and ruthenium not only stirring is crucial, but also an optimized reagent mixture of hydrochloric acid with perchloric acid. Tipps and tricks for sample after-treatment and for clean and comfortable operation and handling are given.

If silicon was not to be determined, complete digestion of up to 500 mg of these PGMs is possible in the sealed quartz vessels. The completeness of the digestion was assessed by optical criteria: no particulate residues in the intensely colored solutions.

(P-10)

Hyphenating HPLC and ICP-MS: the case of polysulfide anions

Aleksei Sadykov[a], Martin Winter[a,b], Simon Wiemers-Meyer[a], Sascha Nowak[a]

[a]*University of Münster, MEET Battery Research Center, Corrensstr. 46, 48149 Münster, Germany*
[b]*Helmholtz-Institute Münster, IEK-12, Forschungszentrum Jülich GmbH, Corrensstr. 46, 48149 Münster, Germany*

Coupling liquid chromatography (LC) to inductively coupled plasma-mass spectrometry (ICP-MS) offers great opportunities for obtaining speciation information in complex mixtures. Nonetheless, using ICP-MS as a detection unit for LC separation faces some challenges such as instability of signal as organic solvents are introduced to the plasma, varied ICP-MS response under the gradient eluent flow, peak broadening due to increased pathway in high-volume sample introduction systems (10–100 mL). Here, we propose a facile LC-ICP-MS method for speciation of polysulfide anions in Li_2S_n mixtures in organic solvents which takes into account all the above-mentioned drawbacks.

First, for a rapid and full derivatization of polysulfide anions in the mixture, an appropriate derivatization agent was selected. Next, the composition of eluent was adjusted for a complete separation of the mixture of derivatized organic polysulfides in the LC setup. Using methanol-water mixture as a low-carbon eluent promoted the stability of plasma while the separation remained at an appropriate level. Also, applying gradient LC mode was proved to be impossible as the ICP response decreased with increasing methanol content.

To optimize the LC-ICP-MS response, the eluent flow rate, sample gas flow and additional O_2 gas flow were adjusted. It was shown that the chromatography peak shape depends on gas parameters and eluent flow. For example, a sharper chromatography signal was achieved when the sample gas flow rate was 0.7 L/min and eluent flow was 0.5 ml/min. It was also revealed that the addition of small amounts of O_2 leads to the increase of signal-to-noise ratio and signal sharpness with no change of the peak width. In addition, it was demonstrated that the peak width can be minimized by using a spray chamber of smaller volume.

The developed method allowed us to quantify the polysulfide distributions Li_2S_n generated in organic solvents with *n* ranging from 4 to 8, with tetrasulfide found to be the most abundant species in all mixtures. The method described may shed light on the polysulfide shuttle effect which occurs in Li-S batteries during gradual reduction of sulfur cathodes and leads to a fast capacity fade of those. The quantitative estimation of polysulfides formed in electrolytes with different additives during cycling can provide insight into using suitable components for this type of batteries.

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(P-11)

Combining full spectrum read-out and innovative automation capabilities for robust and fast analysis using ICP-OES

Mikael Axelsson, Sukanya Sengupta and Daniel Kutscher

Thermo Fisher Scientific, Sweden

Inductively coupled plasma optical emission spectroscopy (ICP-OES) is the technique of choice for elemental analysis, as it provides the ability to measure up to %-levels for certain analytes, in combination with trace level analysis for critical contaminants. A primary area where ICP-OES excels is the analysis of fertilizers, which need to be screened for the correct amount of essential nutrients, as well as the confirmation that potentially harmful or even toxic elements are not introduced into the environment.

Such analysis is typically carried out in testing laboratories that often face a seasonal variation of the sample load, causing an extreme demand on sample throughput and response time. In such situations, shortening the sample analysis time helps to maximize laboratory throughput and conduct sample analysis within the committed timelines. This requires a fast and reliable ICP-OES instrument and an efficient autosampler to handle hundreds of samples daily. Laboratories further resort to discrete sampling valve systems for reducing time spent on sample delivery and successive washing steps. However, such accessories introduce additional complexity to the entire analytical setup and require a significant additional financial investment.

This presentation will highlight how ICP-OES can be used to simultaneously measure the total concentrations of different major and trace elements (including the total concentration of Cr) within fertilizers with high speed and accuracy. This combination of instruments offers unique features that help testing laboratories to increase productivity without compromising robustness, by enabling the use of a simple analysis method that is capable of sample turnover times of less than 40 seconds per sample, without the need to use a discrete sampling valve.

(P-12)

Accurate and reliable multielement analysis of alternative protein foods using ICP-MS

Mikael Axelsson, Sukanya Sengupta and Daniel Kutscher

Thermo Fischer Scientific, Sweden

Plant-based, protein-rich foods and other alternatives to conventional meat and meat-based food products have long been a part of the human diet—termed here as “alternative protein foods.” Historically, foods like soy and other beans, lentils, non-dairy milks like almond milk, locally available edible insects, etc. have been consumed traditionally in many parts of the world and in smaller amounts as “alternative” options elsewhere. In recent times, an exponential increase of the plant-based alternatives has been seen globally due to increasing concerns of the impact of conventional and industrial meat production on the global climate and the environment, as well as ethical concerns and the effects of over-consumption of animal-derived products on human health. The Commission Regulation (EU) 2023/915121, for example, lays down guidelines on permitted levels of contaminants, including toxic metals, in many different foods including some alternative options. For example, the prescribed limits for Cd in pulses and proteins from pulses in this regulation are 0.040 $mg \cdot kg^{-1}$ and 0.10 $mg \cdot kg^{-1}$, respectively, and analytical techniques must be able to accurately determine such low concentration levels in relevant samples.

For a comprehensive multielement analysis of the wide variety of alternative foods, a highly sensitive technology with large linear dynamic range such as inductively coupled plasma mass spectrometry (ICP-MS) is required so low levels of toxic elements as well as high amounts of nutrients like K and Ca can be accurately quantified in the samples.

In this presentation, a method based on single quadrupole ICP-MS has been developed and tested for the accurate, fast, and reliable multielement analysis of ten different alternative protein food samples.

(P-13)

Nano-hydroxyapatite as a novel phosphorus fertilizer

Francesco Minutello, Daniel P. Persson and Søren Husted

University of Copenhagen, Department of Plant and Environmental Sciences, 1871
Frederiksberg C, Denmark.

E-mail: fmi@plen.ku.dk

Traditional phosphorus (P) fertilization practices are marked by considerable inefficiency. When conventional inorganic fertilizers are distributed to agricultural soils, the greatest fraction of the P applied rapidly becomes unavailable to plants due to either chemical fixation or microbial immobilization. Direct application of P to crop leaves can bypass these limitations, but introduces other difficulties, such as potential leaf burn and low uptake efficiency [1].

The integration of nanotechnology in plant science provides the tools for designing smart nanofertilizers capable of addressing these inefficiencies. Since nanoparticles (NPs) possess modifiable physico-chemical surface properties, they can be tailored to deliver nutrients in a variety of chemical forms, offering improvements over conventional mineral salts [2].

Our research explores the efficacy of hydroxyapatite nanoparticles as P fertilizers. In different studies, we apply nano-hydroxyapatite (nHAP) to either the roots [3] or the foliage of P-deficient agricultural crops. Using a combination of techniques to assay P functionality in plant metabolism and bioimaging methods that offer cellular-level resolution, we assess the fertilizing impact of nHAP and investigate mechanisms of interaction between nHAP and plant tissues.
ICP-MS serves as the core method for routine multi-element analysis of both our NPs and plant specimens.

LA-ICP-MS is used in parallel to fluorescence microscopy as a qualitative imaging method to determine the uptake pathways of NPs in roots and leaves, as well as their subsequent translocation or dissolution inside the plant. This approach involves adding trace amounts of rare elements, normally not present in plants, into the structure of NPs, and monitoring their movement inside plants at different time intervals.

To gain further insights into the mechanisms behind NP dissolution and their efficacy as fertilizers, we have also tested LC-ICP-MS to separate and quantify the P incorporated into the NPs from the P released during NP dissolution. While tests on pure NP solutions demonstrate encouraging potential, extending these methodologies to plant samples presents significant challenges. Further efforts are required to overcome these difficulties.

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(P-14)

Optimization of a method for determination of stable boron isotope ratios in various aqueous matrices using multi-collector inductively-coupled plasma mass spectrometry

Julianne Hem[1] and Christian Schöpke[1,2]

[1]*Norwegian University of Life Sciences (NMBU), Ås, Norway*
[2]*Institute for Energy Technology (IFE), Kjeller, Norway*

The chemical element boron (B) has two stable isotopes ^{10}B (19.9%) and ^{11}B (80.1%), with a relative mass difference of 10%. The ratio between the two isotopes has become increasingly important in geochemical and hydrological studies of processes including weathering, transport of pollutants, and as a pH proxy. The large relative mass difference is reflected by a large range of isotopic ratio variability in nature, but also leads to challenges measuring the isotopic ratio without affecting the isotopic ratio in a sample. Accurate measurement of the $^{11/10}B$ ratio via ICP-MS techniques requires understanding of matrix effects, instrumental isotopic fractionation, spectral interferences and other sources of instrumental bias.

In this presentation, we show the results of work performed on the separation of boron from various aqueous matrices (groundwater, freshwater, seawater, landfill leachate and volcanic waters) with a concentration range of B between 10 and 10000 µg/L using the Amberlite IRA743 ion exchange resin on the PrepFAST (ESI, Omaha, USA) automated chromatography system , and measured using a Neptune Plus multi-collector inductively coupled plasma mass spectrometer (Thermo Scientific, Bremen, Germany).We present several of the parameters tested/characterized (separation column lifetime, sample volume, reagent cleanliness, instrumental parameters), as well as results of isotopic characterization on a range of matrices.

(P-15)

Copper speciation in human lung tissue: Comparative analysis between squamous-cell carcinoma and healthy lung tissue using HPLC-ICP-MS

Katarina Marković[1], Aleksandar Stojsavljević[3], Jernej Šribar[4], Igor Križaj[4,2], Radmila Milačič[1,2] and Janez Ščančar[1,2]

[1]*Department of Environmental Sciences, Jožef Stefan Institute, Ljubljana, Slovenia*
[2]*Jožef Stefan International Postgraduate School, Ljubljana, Slovenia*
[3]*Innovation Center, Faculty of Chemistry, University of Belgrade, Belgrade, Serbia*
[4]*Department of Molecular and Biomedical Sciences, Jožef Stefan Institute, Ljubljana, Slovenia*

Copper (Cu) is an essential trace element that plays a crucial role in numerous physiological processes in all living organisms. Within the human body, Cu is present in different chemical species, with the highest concentration, in blood plasma, bound to ceruloplasmin (Cp). Studies show that Cu and Cp concentrations are elevated in serum samples from cancer patients and can potentially be used as biomarkers. Furthermore, Cu accumulation has been observed in various tumor tissues and it is most likely involved in tumor progression.

In the present study, Cu speciation was investigated in squamous-cell carcinoma (SCC) and healthy lung tissues using conjoint liquid chromatography (CLC) on convective interaction media (CIM) monolithic disks coupled with inductively coupled plasma mass spectrometry (ICP-MS). The CLC monolithic column comprised two immunoaffinity (antiHSA) disks and one anion-exchange diethylaminoethyl (DEAE) disk. This setup enabled the separation of Cu bound to Cp, human serum albumin (HSA), and low molecular mass species (LMM) [1].

Tumor samples were homogenized and lysed in the presence of protease inhibitors. Subsequently, the samples were centrifuged for 15 minutes at 12,000 *g*, and the supernatant was subjected to speciation analysis, using CLC on monolithic disks coupled to ICP-MS for the determination of Cu-Cp, Cu-HSA and Cu-LMM species. In addition, Cp was identified using Western blot analysis. To assess the significance of differences in Cu-Cp, Cu-HSA, Cu-LMM, and total Cu concentrations among the selected tissues, a Student’s t-test at a significance level of 0.05 was applied.

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(P-16)

The study of lactoferrin-ruthenium complexes: Insight into the binding mechanism

Tetiana Dyrda-Terniuk[1] and Paweł Pomastowski[1,2]

[1] *Centre for Modern Interdisciplinary Technologies, Nicolaus Copernicus University in Toruń, Wileńska 4, 87-100 Toruń, Poland*
[2] *Department of Inorganic and Coordination Chemistry, Faculty of Chemistry, Nicolaus Copernicus University in Toruń, Gagarina 7, 87-100, Toruń, Poland*

E-mail: tetiana.dyrda-terniuk.@umk.pl (T.D.-T.)

Bovine lactoferrin (bLF), widely known as a multi-functional glycoprotein, shows a high binding affinity to the ferric (III) ions and acts as an iron carrier for several types of cells (immune, intestinal epithelial cells). Due to its unique structure, bLF is capable to bind other metals, including zinc (II), copper (II), chromium (III), manganese (III), cobalt (III), and aluminum (III) ions [1]. The development of metal-protein complexes is an innovative approach that aims to improve existing protein features and to design biomolecules with a wider range of functionality. Recently, the growing trend in studying of ruthenium complexes has been observed which is directly related to their proven anticancer properties. Ruthenium is characterized by less toxicity and causes fewer side effects compared to more widespread in clinical trials platinum complexes (cisplatin, carboplatin, oxaliplatin) [2]. In turn, the much higher selectivity of ruthenium complexes towards cancer cells might be explained by their ability to bind to serum proteins, such as transferrin and albumin [3]. Currently, the information on the ruthenium-lactoferrin interactions is limited. The LF receptors are expressed in numerous cancer cells, including liver, colon, lung, and pancreatic cells. Therefore, the comprehensive investigation of Ru-LF complexes represents a new perspective in targeted cancer therapy. The work aimed to prepare the functionalized Ru-LF complexes and to study the sorption mechanism between ruthenium ions and bLF biomolecule. ICP-OES is considered a crucial instrumental tool for the study of the nature of protein-metal interactions. The quantification of metal binding provided insight into the binding capacity and the stoichiometry of the protein-metal complex. The effect of pH and ionic strength conditions on the ruthenium binding efficiency was evaluated. In turn, the kinetic studies allowed to clarify several steps in the sorption process (surface sorption, intraparticle diffusion) and provided information on the spontaneity of Ru-LF binding. The understanding of Ru-LF interactions with the application of the ICP-OES technique is an essential step for the drug design and for the further analysis of its therapeutic efficiency.

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(P-17)

The impact of production processes on selected heavy metals distribution in dairy products of Kuyavian-Pomerian Voivodeship, Poland

Oleksandra Pryshchepa[1] and Paweł Piotr Pomastowski[1,2]

[1]Centre for Modern Interdisciplinary Technologies Nicolaus Copernicus University in Torun, Wileńska 4, 87-100 Toru, Poland
[2]Department of Inorganic and Coordination Chemistry, Faculty of Chemistry, Nicolaus Copernicus University in Torun, Gagarina 7, 87-100, Torun, Poland

Milk, being a liquid wholesome food for newborns, is characterized by a complex chemical composition rich in essential nutrients such as proteins, fats, carbohydrates, minerals, and vitamins. Despite the health benefits that dairy products provide, they may also contain residues of various contaminants, such as toxic heavy metals from the environment. Heavy metals is a broad term used to describe a diverse group of elements characterized by relatively high densities, atomic weights, or atomic numbers. In the context of EU regulations concerning food contaminants, heavy metals typically include lead, cadmium, mercury, inorganic tin, and arsenicdue to their significant cytotoxic properties. Primarily, heavy metals enter milk as a consequence of their consumption by animals through feed and water. However, the manufacturing process of dairy products is a multi-step process aimed at extending the storage period of these valuable resources, but it can also lead to unwanted contaminations. It's worth noting that milk tanks and all production machines are typically made of stainless steel, mainly maid from iron and chromium. Understanding the content of metals in dairy products is critical due to their varying impact on human health. Toxic heavy metals, characterized by their high affinity for proteins, pose significant health risks. Given that milk is rich in proteins, it can inadvertently facilitate the mobilization of these metals from the production equipment into the dairy products themselves. This is not only a concern for the safety and quality of milk but also affects the distribution of heavy metals across a range of dairy products. Consequently, assessing the presence and concentration of heavy metals in dairy is paramount to ensuring consumer safety and complying with health regulations. Our study aimed to comprehensively analyze the heavy metal content in milk and dairy products produced by a local dairy factory in the Kuyavian-Pomeranian Voivodeship, Poland. By employing the Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) technique, we targeted the detection and quantification of five toxic heavy metals: cadmium (Cd), mercury (Hg), lead (Pb), arsenic (As), and chromium (Cr). We also measured levels of essential microelements crucial for various biological functions and contribute to the nutritional value of dairy products: magnesium (Mg), calcium (Ca), sodium (Na), potassium (K), iron (Fe), and zinc (Zn), which are crucial for various biological functions and contribute to the nutritional value of dairy products. This dual focus not only highlights the potential hazards associated with heavy metal contamination but also underscores the importance of essential microelements in maintaining the nutritional integrity of dairy products.

The study was financially supported by the Agency for Restructuring and Modernization of Agriculture(Poland), within the Rural Development Program for 2014-2020, Measure M16 "Cooperation", project Nr.00126.DDD.6509.00244.2022.02, titled “*Development of a new technology for the production of beta butter with health-promoting properties*”.
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(P-18)

Trace element analysis of pegmatite lithium ores by GD-MS

Gideon Bartov and Andrew M. Zipkin

Eurofins EAG Laboratories, 103 Commerce Blvd., Liverpool NY 13088, USA

The lithium-ion battery (LIB) industry sources high purity lithium carbonate and lithium hydroxide from lithium-rich brines and "hard rock" lithium ore. Hard rock lithium is mainly found in spodumene and petalite – lithium-aluminum-silicates found in pegmatite deposits. As the world shifts to more sustainable and equitable mining, it is important to be able to differentiate among ore sources to ensure supply chain integrity. Different deposits have potentially unique ratios of trace element concentrations which can serve as "fingerprints" for source discrimination and traceability of lithium products. Furthermore, battery manufacturers are demanding increasingly stringent purity requirements for lithium carbonate and lithium hydroxide products manufactured from hard rock lithium. GDMS is positioned to expand the list of measurable trace impurities beyond the limited set traditionally measured by ICP-OES.

Typically, lithium ore is analyzed using one or more of the following approaches: ICP-OES, ICP-MS, and XRF. The major weakness of these techniques is sample preparation. It is time-consuming, has the potential to introduce contamination into the sample, and generates hazardous waste. XRF is widely used to analyze rocks and sediments but is unable to reach sub-ppm sensitivity.

Glow Discharge Mass Spectrometry (GDMS) is a solid-state sampling technique that is well-positioned to perform fast, reliable analysis of lithium ores and concentrates for trace element impurities. GDMS does not require sample prep and only uses a small amount (~10-20 mg) of material. It can analyze the full mass range – excluding H, C, N, O – and is able to reach sub-ppm sensitivity. While it is a semi-quantitative technique, we apply relative sensitivity factors (RSFs) to account for ion yields in the plasma, ensuring a more accurate concentration measurement. Semi-quantitative concentration data from techniques like XRF and LA-ICP-MS are widely used in geochemistry and archaeometry for source discrimination studies of geomaterials. GDMS is positioned to improve on this approach by utilizing oxide-specific RSF values to obtain more accurate concentrations.

We analyzed a suite of commercially available lithium ore standards from OREAS (OREAS 750, 751, 752, 753, 999) which were then processed into nanopowders by myStandards GmbH. We analyzed the powders using an Ametek Nu Instruments Astrum GDMS. Using an oxide RSF set developed in-house, we were able to obtain consistent results across multiple days of analysis. Out of 54 analyzed elements, including elements with indicative values, 10 were within 10% recovery, 19 were within 25% recovery, and 47 were within a factor of two of the certificates – the expected methodological precision range for GDMS. More importantly, the RSD of the measured elements across all 5 OREAS powders were generally better than 25%, with 45 of the 54 elements exhibiting less than 25% RSD variations between standard powders. This long-term consistency is vitally important when comparing element ratios for source discrimination and traceability.

GDMS is poised to provide a fast and reliable analytical solution to the increasing demand for traceability and purity requirements from LIB producers.

(P-19)

Advancing stable and long-lived radiogenic isotope ratio measurements of environmental pollutants

Tea Zuliani[1], Dmitriy Malinovskiy[2], Violeta Hansen[3], Rasmus Andreasen[4], Jochen Vogl[5], Pranav Seena Prem[5], Jixin Qiao[6], Daniel Pröfrock[7], Dominik Wipperman[7], Christian Schöpke[8], Johanna Noireaux[9], Clemens Walther[10], Aaron Lehnert[10], Johanna Irrgeher[11], Shaun T. Lancaster[11], Stefan Wagner[11], Emma Braysher[12], Janine Eberhardt[13], Lukas Flierl[13], Betül Arı Engin[14] and Dirk Arnold[13]

[1]*Jožef Stefan Institute, Department of Environmental Sciences, Ljubljana, Slovenia,*
[2]*National Measurement Laboratory, LGC, Queens Road, Teddington, UK*
[3]*Gothenburg University, Department of Radiation Physics, Sahlgrenska University Hospital, Sweden*
[4]*Department of Geoscience, Aarhus University, Denmark*
[5]*Bundesanstalt für Materialforschung und –prüfung (BAM), Division of Inorganic Trace Analysis, Berlin, Germany*
[6]*Department of Environmental and Resource Engineering, Technical University of Denmark, Roskilde, Denmark*
[7]*Helmholtz Zentrum Hereon, Institute of Coastal Environmental Chemistry, Geesthacht, Germany*
[8]*Institute for Energy Technology, IFE, Kjeller, Norway*
[9]*Laboratoire national de métrologie et d'essais (LNE), Paris, France*
[10]*Institute of Radioecology and Radiation Protection, Leibniz University Hannover, Hannover, Germany*
[11]*Montanuniversität Leoben, Department of General, Analytical and Physical Chemistry, Leoben, Austria*
[12]*National Physical Laboratory, Hampton Road, Teddington, UK*
[13]*Physikalisch-Technische Bundesanstalt, Bundesallee 100, Braunschweig, Germany*
[14]*TÜBİTAK National Metrology Institute, 41470, Gebze/Kocaeli, Türkiye*

The isotopic compositions of elements vary in the environment due to processes such as radioactive decay, cosmic ray spallation, mass-dependent and mass-independent fractionation, and different anthropogenic sources such as industrial activities, nuclear weapon testing, nuclear accidents, as well as natural phenomena long-range atmospheric and water transport, climate change, etc. Isotope ratio data obtained by mass spectrometry now plays an important role as tracers of a multitude of processes in Earth and environmental sciences.

Inductively coupled plasma mass spectrometry (ICP-MS) is a widely used technique for detecting variations in isotopic abundances in e.g., environmental samples. Recent advancements in mass spectrometric instrumentation have significantly enhanced sensitivity and precision, enabling the reliable detection of even minute variations in the isotopic composition of most elements with more than one natural isotope. However, to fully realize these new capabilities, a significant analytical challenge remains due to a vast number of analyte/matrix combinations, concomitant matrix effects, spectral interferences, and instrumental isotopic fractionation that must be characterized before and during the measurements. Success in developing new and improved methods for stable and long-lived radiogenic isotope ratio measurements by mass spectrometric methods depends on improvements in the reduction and quantification of uncertainties associated with sample preparation, instrumentation, and external calibration.

The goal of this presentation is to show the key findings of the European Partnership on Metrology project “Metrology for the harmonisation of measurements of environmental pollutants in Europe” - MetroPOEM (https://www.npl.co.uk/euramet/metropoem), with a focus on the ICP-MS techniques used for isotope ratio measurements. Several elements were used as model systems for the development and optimisation of sample preparation and measurement procedures, including Li, B, Cr, Cd, Ni, Sb, Pb, and U. Isotope ratios of these elements determined by multi-collector ICP-MS, sector field ICP-MS, quadrupole ICP-MS, and ICP-MS/MS will be assessed and compared. Data on their performance will be presented and assessed in terms of accuracy and precision. Comprehensive uncertainty budgets for the determination of the isotope ratios of the selected elements by different calibration approaches will be presented. The presented results will highlight the potential of recent advances in ICP-MS technology.

(P-20)

Determination of critical raw materials in micromobility e-vehicles by ICP-OES

Riikka Turkki, Siiri Perämäki and Ari Väisänen

Department of Chemistry, University of Jyväskylä, Finland

The number of micromobility and thus also the number of vehicles used for micromobility, such as e-scooters and e-bikes, is increasing. As the number and types of vehicles increase, so does the need for batteries, magnets, and printed circuit boards. These components contain valuable metals the most of which are classified as critical raw materials (CRMs). The determination of element concentrations was performed on the black mass of Li-ion batteries and NdFeB magnets of various e-vehicles, such as e-scooters, e-bike and e- hoverboard. In research and the review of the analysis results of the samples, an effort was made to consider the differences between e-scooters in private use and e-scooters in public rental use. The black mass and magnets of e-vehicles were mechanically pretreated, and microwave digested using either aqua regia or reverse aqua regia. The determination of element concentrations from dissolved samples was performed with ICP-OES.

The elemental concentration determined of the e-vehicle black mass samples showed that there are significant differences in the proportions, for example, of nickel (~10-25 wt%), manganese (~0,2-20 wt%) and copper (1-20 wt%). The lithium, cobalt, and antimony, on the other hand are present at same level of concentration in all e-vehicle batteries investigated. In the case of magnets, the differences were significant in terms of neodymium and cerium concentrations. The iron concentrations resulted in over 60% for all the magnets of e-vehicles investigated. The magnets also contained low concentrations of other rare earth elements (REEs) such as praseodymium, samarium, and gadolinium.

(P-21)

Elemental analysis of mango flesh and peel by ICP-MS

Michaela Zeiner[1], Ema Mihalić[2], Iva Juranović Cindrić[2], Ivan Nemet[2] and Heidelore Fiedler[1]

[1]*Örebro University, School of Science and Technology, Man-Technology-Environment Research Centre, Fakultetsgatan 1, 70182, Örebro, Sweden*
[2]*Sveučilište u Zagrebu Prirodoslovno-matematički fakultet, Kemijski odsjek, Zavod za analitičku kemiju, Horvatovac 102a, 10000 Zagreb, Hrvatska*

This study focused on the elemental analysis of commercially available dried mango, fresh mango and its peel. The general method consisted of microwave-assisted digestion followed by quantification of the analytes through inductively coupled mass spectrometry (ICP-MS). Four different digestion mixtures (nitric acid and hydrogen peroxide) were tested. Using statistical tools (Student's test) and chemometric analysis (principal component analysis, PCA), the results obtained via different digestion procedures were compared. It was found that there only concentrated nitric acid less efficient than combinations of nitric acid and hydrogen peroxide for the given plant material.

The accuracy of the method was verified with certified reference materials (NIST SRM 1547 – *Peach leaves* and NIST SRM 1573a - *Tomato Leaves*).
The most abundant elements in all samples were macroelements: K, Ca, Mg and Na. Regarding the essential microelements, Mn, Cu and Zn were most abundant. No significant difference in the mass fractions of the elements between the dried mango samples were found except for Na content, which is significantly higher in one sample. Fresh mango contained more Ca than commercially available dried mango samples, while the mass fractions of other macroelements are similar. The most abundant trace element in commercially available dried mango samples was Rb, while it was found in smaller amounts in the self-dried sample. All samples had mass fractions of Cd and Pb below the respective permitted values given by the European Food Safety Authority of these heavy metals in food, namely 0.05 mg/kg and 0.1 mg/kg.

Comparing the dried fresh mango and its peel revealed that mango peel after drying contains statistically significantly higher contents of Ca, Mg, Mn, and Sr. The most abundant potentially toxic element in all samples was Al, with the highest mass fraction in the peel.

In concluding dried mango is a good source of essential elements, especially Ca, K, Mg and Mn. Toxic elements such as Cd, Pb, As, which would pose a danger to human health, were not found in contents above the permissible level. The small amount of dried mango, which can significantly contribute to the recommended daily intake (RDA) of essential elements, and the high nutritional value make mango an excellent and healthy snack.

(P-22)

Analysis of plant material from phytoremediation processes of soils contaminated with lithium ion battery materials using plasma-based methods

Julius Buchmann[a], Martin Winter[a,b], Simon Wiemers-Meyer[a] and Sascha Nowak[a]

[a]*University of Münster, Münster Electrochemical Energy Technology (MEET), Corrensstraße 46, 48149 Münster, Germany*
[b] *Helmholtz-Institute Münster (HIMS), IEK-12, Forschungszentrum Jülich, Corrensstraße 46, 48149 Münster, Germany*

Lithium ion batteries (LIBs) are a key technology for decarbonization of the energy industry and the mobility sector, *e.g.* as stationary storage and in electric vehicles. However, current state-of-the-art LIBs rely on critical raw materials such as lithium, nickel and cobalt.[1] Low environmental standards in mining areas and a lack of closed recycling loops, respectively, inadequate disposal of batteries and electronic consumer devices, pose the threat of battery materials being liberated into the environment.[2] Potentially toxic metals such as nickel or cobalt can be dispersed and accumulated in the environment.[3] The concept of phytoremediation uses plants which are able to extract metals from soils into their upper biomass, which represents a less invasive, sustainable and economic method for remediating contaminated soils.

The phytoremediation potential of brown mustard (*brassica juncea*) was explored in a pot study by cultivation of plants in soil contaminated with LIB materials. By using ionic metal solutions and solid battery material contaminations, the overall accumulation potential of brown mustard for these metals and the interdependence of solubility and bioavailability of the metal species could be demonstrated. The metal accumulation in the upper biomass of the plants was investigated by inductively coupled plasma-optical emission spectroscopy (ICP-OES) with liquid sample introduction. Leaf and stem samples were prepared by microwave-assisted acid digestion in *aqua regia*.

The elaborate sample preparation using acid digestion for liquid injection proved to be a bottleneck for large sample numbers. Electrothermal vaporization (ETV)-ICP-OES offers the potential for elemental quantification in solid samples. Therefore, the sample is heated in a graphite furnace while introducing a halogenated reaction gas and transported into the plasma *via* a gas flow. The method was explored to be used for future investigations of plant material contaminated with LIB battery materials, considering matrix effects and applying internal standardization.

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(P-23)

Recovery and ICP-OES analyses for selected metals from electronic waste and rechargeable batteries

Antti Tiihonen[1], Jutta Koskinen[1], Siiri Perämäki[1], Janne Frimodig[1], Jimi Siljanto[1], Joona Rajahalme[2], Sylva Larsson[2], Elmeri Lahtinen[2], Jannatul Rumky[1], Joni Niskanen[2], Roshan Budhathoki[2], Manu Lahtinen[1], Matti Haukka[1] and Ari Väisänen[1]

[1] Department of Chemistry, P.O. Box 35 FIN-40014 University of Jyväskylä, Finland
[2] Formerly University of Jyväskylä, currently affiliated in various industrial sectors

E-mail: antti.j.tiihonen@jyu.fi

Center of Expertise for Circular Economy (CECE) in the University of Jyväskylä Department of Chemistry has concentrated its efforts for research and development of recovery methods and analytical approaches for valuable metals extracted from electronic waste and spent rechargeable batteries. A process to extract pure metals (e.g., Cu, Au) from printed circuit boards (PCBs) has been successfully designed and tested in our mini-pilot scale hydrometallurgical laboratory, waiting for transition to industrial scale application.

Similarly, emerging methods of metal extraction and analyses for nickel, cobalt and rare earth elements from NiMH batteries and magnets has been studied with very efficient dissolution in aqueous media.[1,2]

As the latest cornerstone, research involving individually picked components from PCBs has been studied and an efficient recovery method of tantalum from capacitors has been found using specific zeolite materials.[3]

Gallium extraction from light emitting diodes is currently underway. All research has relied on elemental analyses in aqueous media, specifically conducted with inductively coupled plasma - optical emission spectrometry (ICP-OES) and mass spectrometry (ICP-MS). Due to the complex nature of samples from batteries and PCBs, elemental determinations have been done with different equipment as has been suitable, generally with high throughput and accuracy.

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(P-24)

Understanding water bodies in and around Romerike region in Norway through strontium isotope analysis using MC-ICP-MS

Vani N. Devegowda and Christian Schöpke

Institute for Energy Technology (IFE), Kjeller, Norway

Determination of the stable strontium isotopic composition (^{84}Sr, ^{86}Sr, ^{87}Sr, and ^{88}Sr, and in particular the ratio of radiogenic ^{87}Sr to primordial ^{86}Sr) has emerged as a useful tool in various fields, including environmental science, geology, and archaeology. The radiogenic nature of ^{87}Sr and large local variations in the ^{87}Sr/^{86}Sr ratio in Norway due to varying geological compositions can provide valuable information about the geological source of the strontium and its interactions within the environment.

In this study, we collected water samples from a range of local rivers and other water bodies in and around Romerike region from 2022 to 2023. The collected samples were analyzed for the strontium ^{87}Sr/^{86}Sr ratio via multicollector ICP-MS (Neptune Plus, Thermo Scientific). Prior to analysis, the samples were filtered, and the matrix elements were removed via ESI prepFAST using a CF-MC-SrCa-1000 ion exchange column for an improved measurement accuracy and precision. Our results provide insights into environmental processes and can contribute to a better understanding of naturogenic and anthropogenic inputs to local water bodies.

In addition, we present the results of strontium isotope ratio analysis of the same water bodies before and after the extreme weather event Hans in 2023. We collected samples from affected areas both pre- and post- event. We observed modest changes in Sr isotope ratios after flooding that may have redistributed sediments and materials, potentially impacting the isotopic composition of surface water. We infer that these results may contribute to understanding the effects of future extreme weather events in changing climatic conditions.

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Analysis of petroleum products according to ASTM Method D8110-17

Ewa Pruszkowski [1], Chady Stephan [1] and Daniel Fliegel [2]

PerkinElmer Inc., USA and Canada
[2]PerkinElmer Norway AS, Oslo, Norway

Petroleum, a naturally occurring mixture of hydrocarbons, is found in geological formations and includes not only crude oil but also all liquid, gaseous, and solid hydrocarbons. Under surface pressure and temperature conditions, lighter hydrocarbons exist as gases, while heavier ones are in the form of liquids or solids.

Petroleum products, which include transportation fuels, fuel oils for heating and electricity generation, asphalt and road oil, and feedstocks for making chemicals, plastics, and synthetic materials, are in nearly everything we use.

This abstract presents an analysis of petroleum products using the NexION 2200 ICP-MS, as per the ASTM D8110-17 method, “Standard Test Method for Elemental Analysis of Distillate Products by ICP-MS”. This method describes the procedure for determining a few trace elements in light and middle distillate petroleum products.

The results demonstrate the NexION 2200 ICP-MS’s capability to effectively analyze petroleum distillates according to the ASTM D8110-17 method requirements, providing the outstanding performance needed for the petroleum industry when analyzing difficult samples. This work underscores the importance of advanced analytical techniques in ensuring the quality and safety of petroleum products.

(P-26)

The analysis of drinking water using universal collision-reaction gas technology on the NexION 1100 ICP-MS

Pritesh Patel[1] and Zuo, Minyu[2]

[1]PerkinElmer Inc., USA and Canada
[2]PerkinElmer, Stockholm, Sweden

Drinking water, primarily sourced from rivers, lakes, and groundwater, contains varying concentrations of elements influenced by the underlying geology and anthropogenic activities in catchment areas. With increasing urbanization, industrialization, mining, and farming practices, the need to monitor potable water for hazardous components has become crucial. Many countries have implemented stringent criteria that must be met before water can be distributed for human use.

The new NexION 1100 ICP-MS has proven to exceed expectations for low detection limit requirements in potable drinking water standards globally, using a single, universal collision-reaction gas. Its efficacy is particularly notable in analysing trace concentrations of 78 Se with MDLs lower than 10 ppt, a task often considered challenging due to its low ionization efficiency and natural abundance.

The method’s accuracy and robustness have been validated through the analysis of certified reference materials and spiked tap water samples. Its stability was demonstrated over 13 hours using a single calibration. The new NexION 1100 ICP-MS, with its unique Universal Collision-Reaction Gas Technology, promotes productivity while achieving low ppt detection limits, meeting the requirements of many high-throughput laboratories.

(P-27)

Enhancing trace element determination in seawater using NexION ICP-MS systems

Liyan Xing[1], Ewa Pruszkowski[1], Chady Stephan[1], Aaron Hineman[1] and Daniel Fliegel[2]

[1]PerkinElmer U.S. LLC
[2]PerkinElmer Norway AS, Oslo, Norway

The analysis of trace elements in seawater presents a significant challenge for inductively coupled plasma mass spectrometry (ICP-MS) due to the high total dissolved solids (TDS) content in this matrix. The elevated concentrations of matrix components, including sodium, magnesium, and chloride ions, can lead to polyatomic spectral interferences, complicating the accurate determination of elements such as As, Co, V, Zn, Cu, and Fe. Even for elements less affected by spectral interferences, such as Cd, Sn, and Pb, their low parts-per-trillion (ppt) concentrations in seawater pose precision and accuracy difficulties.

We investigate two advanced ICP-MS systems: the NexION® 2200 and the NexION® 5000 Multi-Quadrupole ICP-MS. Both instruments are equipped with the Universal Cell Technical (UCT), allowing for flexible analysis modes, including Standard, Collision with Kinetic Energy Discrimination (KED), and Reaction with Dynamic Bandpass Tuning (DBT).

Key Findings:

1. **Exceptional Performance:** Both NexION systems demonstrate superior interference removal capability and outstanding sensitivity. Exceptionally low detection limits (single to double digits in ppt) were achieved for most trace elements.
2. **Method Validation:** The accuracy of the method was validated through recoveries of seawater certified reference materials (CRMs) and standard spiked seawater samples. Excellent recoveries within ±10% were obtained for most analytes on both instruments.

Our work showcases the robustness of direct seawater analysis using NexION ICP-MS systems, supported by PerkinElmer’s All Matrix Solution (AMS) and High Throughput System (HTS). Researchers and environmental analysts can confidently utilize these systems for precise and reliable trace element measurements in challenging seawater matrices.

(P-28)

Investigation of the charge state of lithium ion battery active materials by means of single particle ICP-OES

Marc Vahnstiege[1], Alexandros Tsoufios[1], Martin Winter[1,2], Sascha Nowak[1] and Simon Wiemers-Meyer[1]

[1]University of Münster, MEET Battery Research Center, Corrensstraße 46, 48149 Münster, Germany
[2] Helmholtz-Institute Münster, IEK-12, FZ Jülich, Corrensstraße 46, 48149 Münster, Germany

The lithium ion battery (LIB) is the most widely implemented electrochemical energy storage system in the world, used in a multitude of applications like smartphones, notebooks, electric vehicles and many more. Consequently, large efforts in scientific research have been made to increase the energy density, cycle as well as calendar life and reduce potential safety hazards of this cell system. Therefore, it is essential to gain an understanding of the redox processes of the cathode active material (CAM) that occur during operation of the battery cell. At the nano- to microscale of the CAM, local inhomogeneities of the electrode, properties of the active material and aging phenomena can influence such redox processes. Consequently, local inhomogeneities in the state-of-charge (SOC) can emerge, affecting the capacity of the cell and accelerating the degradation of the active material.

To quantify the SOC of the active material, single particle inductively coupled plasma optical emission spectroscopy (SP-ICP-OES) can be utilized for the elemental analysis of individual CAM particles.[1] In doing so, lithium and the respective transitions metals can be analyzed for each particle that is introduced into the plasma. Based on the measured intensity of the respective elements, the intensity ratio of lithium to the respective transition metal be calculated. This can be used to quantify the lithiation degree in order to investigate the charge state distribution of the active material particles.

In the study presented on this poster, nickel-rich lithium transition metal oxides with the general formula of $Li[Ni_{0.8}Mn_{0.1}Co_{0.1}]O_2$ are used. For this kind of active material, the Li 670.776 nm to Ni 231.604 nm intensity ratio of the analyzed particles can be determined. To then estimate the SOC of the particles, a matrix-matched external calibration is used. Therefore, the CAM is delithiated to approx. 20%, 40%, 60% and 80% SOC, resulting in decreasing Li/Ni intensity ratios as the charge state increases. In addition to that, electrochemical aged cells were analyzed to identify heterogeneities in the SOC distribution of the particles after cycling.

[1] T.-N. Kröger, S. Wiemers-Meyer, P. Harte, M. Winter and S. Nowak, *Analytical Chem*istry, 2021, **93**, 7532-7539.

(P-29)

Determination of elements and element species in size resolved urban aerosol samples by HPLC-ICP-MS/MS

Stefan Tanda[1], Balázs Berlinger[2] and Walter Gössler[1]

[1]Department of Chemistry, University of Graz, Universitätsplatz 3, 8010, Graz, Austria
[2]Department of Animal Hygiene, Herd Health and Mobile Clinic, University of Veterinary Medicine, István u. 2., 1078, Budapest, Hungary

E-mail: berlinger.balazs@univet.hu

Extensive information is available on total arsenic in particulate matter (PM), but little is known about the relative contribution of each individual species.
In the present study size resolved urban aerosol samples were analysed with state-of-the-art analysis techniques including high performance liquid chromatography (HPLC) coupled to inductively coupled plasma mass spectrometry (ICPMS). In order to gain useful information on concentration levels of different elements (mostly metals) and some of their species in urban aerosol size fractions; elements: such as lithium (Li), sodium (Na), magnesium (Mg), aluminium (Al), potassium (K), calcium (Ca), phosphorus (P), sulphur (S), vanadium (V), chromium (Cr), manganese (Mn), iron (Fe), cobalt (Co), nickel (Ni), copper (Cu), zinc (Zn), rubidium (Rb), strontium (Sr), arsenic (As), molybdenum (Mo), selenium (Se), cadmium (Cd), tin (Sn), antimony (Sb), tungsten (W), thallium (Tl), lead (Pb), bismuth (Bi) and uranium (U), as well as some arsenic species were determined in the PM size fractions.

Urban aerosol samples were collected in a winter and a summer period in Oslo, Norway. A Model 125R NanoMOUDI-IITM (Microorifice Uniform Deposit Impactor; MSP corporation, TSI, Shoreview, MN, USA) was used for the collection. The impactor was equipped with 5 µm pore size polycarbonate (PC) foils (Merck Millipore, Burlington, MA, USA). PM loaded foils were cut in two halves. 50% of each PC foil was used to determine total elemental content. The other half of each filter was used to quantify arsenic species. For total element determination, foils were digested in an ultraCLAVE III microwave digestion system (MLS GmbH Mikrowellen-Labor-Systeme). Furthermore, Standard Reference Material (SRM) 1648a Urban Particulate Matter (National Institute of Standards & Technology), a set of digestion blanks and a set of foil blanks were digested with each set of samples. For the determination of organoarsenicals, namely trimethylarsine oxide (TMAO), dimethylarsinate (DMA), methylarsonate (MA), and total inorganic arsenic as arsenate a combined extraction and oxidation technique was used. An Agilent 8900 triple quadrupole ICPMS/MS (Agilent Technologies, Santa Clara, CA, USA) was used for the determination of total element concentrations. An HPLC system was coupled to the ICPMS/MS for the arsenic speciation analysis [1].

Information on elemental concentration levels and the distribution of these elements between different urban aerosol size fractions are presented on the conference. The information on the distribution of elements (and element species) allows to have a better estimate of the dose of elements, which possibly deposit in the human airways after being exposed to urban air. Furthermore, regarding arsenic speciation analysis, the obtained data might give new insights into the topic of arsenic biovolatilization.

This work was supported by the National Research, Development and Innovation Fund under grant 2021-1.2.4-TÉT-2021-00001.

[1] Tanda, S., Gingl, K., Licbinsky, R., et al. (2020) Environ. Sci. Technol. 54, 5532-5539.

(P-30)

Elemental and isotopic analysis of aged honeys – A North American perspective on unique sweeteners

Ágota Zsófia RAGYÁK[a], Tamás VARGA[b], Nassar Almonther Khaled AHMED[a], Mihály BRAUN[b], Anikó HORVÁTH[b], László PALCSU[b], Zsuzsa LISZTES-SZABÓ [b], Mihály MOLNÁR[b], A.J. Timothy JULL[b,c,d], Szilárd SZABÓ[e], Krisztina Hajduné KOSDI[b], István FUTÓ[b] Jim KASTE[f], Edina BARANYAI[a], Zsófi SAJTOS[a]

[a] Environmental Analytical Research Group, Department of Inorganic and Analytical Chemistry, Faculty of Science and Technology, University of Debrecen, Egyetem Square 1, H-4032 Debrecen, Hungary
[b] International Radiocarbon AMS Competence and Training (INTERACT) Center, HUN-REN Institute for Nuclear Research, Debrecen, H-4026, Bem Square 18/c, Hungary
[c] Department of Geosciences, University of Arizona, Tucson, AZ 85721 USA
[d]University of Arizona AMS Laboratory, Tucson AZ 85721 USA
[e]Department of Physical Geography and Geoinformatics, Faculty of Science and Technology, University of Debrecen, Debrecen, Hungary
[f] Geology Department, William & Mary, Williamsburg, VA, USA

Honey, renowned for its natural sweetness and versatility, serves as a flavorful and healthier alternative to traditional sweeteners in various culinary applications. This worldwide used sweetener, however, is extremely affected by anthropogenic activity through extensive agriculture, industrialization or traffic, and the impact of these influences can be detected by element and isotope analytical tools. Our former studies on unique, old series of EU (Hungarian) honeys proved their excellent relevance in reflecting the environmental conditions over a longer period of time based on their inorganic constituents but also showed not-contemporary carbon contribution which amount looks random, varying honey to honey. In present work the elemental composition and the contribution of the old carbon in aged honey samples from the eastern part of the USA were analyzed to verify the previous observations (in the EU) in a very different environment and continent. We used modern isotope ratio measurements ($^{13}C/^{12}C$, $^{14}C/^{12}C$,) to determine the carbon isotopic composition and radiocarbon age of different US honey samples as well as the elemental concentration, Sr and Pb isotope ratio of the received honeys was determined by different inductively coupled plasma techniques (ICP-OES, ICP-MS, and MC-ICP-MS)

The multivariate statistical analysis of the elemental content shows a separation based on both the botanical origin of the nectar producing species and on the geographical origin of the nectar producing sites. A slight latitudinal effect was observed by the $^{13}C/^{12}C$ isotope ratio analysis, but honey samples from the Eastern part of the USA showed significant, 50% 10 years or even older carbon contribution to the honey by the radiocarbon analysis; that effect is random and does not have a correlation with the stable isotope signature. In specific cases, the measured values were even 20 ‰ ($\Delta^{14}C$) higher than the expected atmospheric $^{14}CO_2$ value at the given year, which shows great old carbon contribution, presumably from not nectar origin excreted plant liquids, which shifts the calibrated age of the sample by even more than 5 years. Samples where adulteration is assumed based on the isotope analytical results show no statistically significant difference either in the composition or the concentration of the inorganic components.

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Quantitative imaging of a lunar meteorite sample by laser-induced breakdown spectroscopy trained on energy dispersive spectroscopy data

Daniel Holub[1,2], Timon Schild[3,4], Dennis Harries[3,5], Pavel Pořízka[2,6] and Jozef Kaiser[1,2,6]

[1] Faculty of Mechanical Engineering, Brno University of Technology, Technická 2896, 616 69, Brno, Czech Republic
[2] CEITEC Brno University of Technology, Purkyňova 123, 612 00, Brno, Czech Republic
[3] European Space Resources Innovation Centre, 5, Avenue des Hauts-Fourneaux, L-4362, Esch-sur-Alzette, Luxembourg
[4] University of Luxembourg, 2 Av. de l'Universite, 4365, Esch-Belval, Esch-sur-Alzette, Luxembourg
[5] Luxembourg Institute of Science and Technology, 5 avenue des Hauts-Fourneaux, L-4362, Esch-sur-Alzette, Luxembourg
[6] Lightigo s.r.o., Renneská třída 329/13, 639 00 Brno – Štýřice, Czech Republic
E-mail: Daniel.holub@vutbr.cz

With the impending depletion of Earth-bound natural resources and renewed interest in extraterrestrial human activities, the focus is now on the closest extraterrestrial body – the Moon. In the fast-growing industry of In-Situ Resource Utilization (ISRU) for lunar resources, robust, reliable, and rapid assessment of the quality of resources is needed. The knowledge of the composition of the lunar regolith comes not only from an in-situ analysis, but also from lunar material either delivered to Earth by humans (Apollo samples), or in the form of lunar meteorites. These samples are rare, but any proposed methodology can be proven and tested in laboratory settings.

In this project, a lunar meteorite (Northwest Africa – NWA – 10203) recovered in the Sahara desert has been analyzed by Laser-Induced Breakdown Spectroscopy (LIBS) and Energy Dispersive Spectroscopy (EDX) to assess the mineralogical composition of the meteorite. By using large-scale LIBS with good resolution (20 μm), the whole meteorite area (22×15 mm) could be evaluated, yielding more than 1 million spectra per sample. By assessing the elemental spatial distribution, two areas of approximately 4×2 mm were selected for the EDX measurements.

By a data analysis pipeline including masking, standardization, normalization, Principal Component Analysis (PCA), clustering, and data fusion the data from both methods was combined to provide quantitative elemental distribution maps for the whole surface of the sample. These results were then analyzed from the mineralogical point of view to identify minerals present in the sample. The abundance of these minerals could then be determined on the basis of the combined data. Using this methodology, quantitative information can be gained by LIBS measurements on a wider range of samples without the need for lengthy and tedious sample preparation needed for other methods such as EDX.

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Dental ankylosis' developmental stages determination by correlative imaging and data fusion

Anna Konečná[1], Daniel Holub[1,2], Jan Štembírek[3,4], Pavel Hurník[4,5,6], Marcela Buchtová[4,7], Pavel Pořízka[1,2,8] and Jozef Kaiser[1,2,8]

[1] CEITEC Brno University of Technology, Purkyňova 123, 612 00, Brno, Czech Republic
[2] Faculty of Mechanical Engineering, Brno University of Technology, Technická 2896/2, 616 69, Brno, Czech Republic
[3] Department of Oral and Maxillofacial Surgery, University Hospital Ostrava, 17. listopadu 1790/5, 708 52 Ostrava-Poruba, Czech Republic
[4] Laboratory of Molecular Morphogenesis, Institute of Animal Physiology and Genetics, Czech Academy of Sciences, Veveří 967/97, 602 00 Brno, Czech Republic
[5] Institute of Molecular and Clinical Pathology and Medical Genetics, University Hospital Ostrava, 17. listopadu 1790/5, 708 52 Ostrava-Poruba, Czech Republic
[6] Institute of Molecular and Clinical Pathology and Medical Genetics, Faculty of Medicine, University of Ostrava, Syllabova 19, 703 00 Ostrava, Czech Republic
[7] Department of Experimental Biology, Masaryk University, Brno, Czech Republic
[8] Lightigo s.r.o., Renneská třída 329/13, 639 00 Brno – Štýřice, Czech Republic
E-mail:Daniel.holub@vutbr.cz

Tooth ankylosis is a medical term referring to a fusion between a tooth and underlying bony support tissue. While it is natural for some species, human teeth ankylosis is considered pathological and usually occurs after a trauma or prolonged inflammation. The range of symptoms of human teeth ankylosis is wide – from completely asymptomatic to severe functional and aesthetic problems. As the removal of the ankylotic tooth is often connected to extensive surrounding bone loss, the need for a more in-depth understanding of the pathology is critical for enabling earlier diagnosis. So far, the diagnosis is usually done according to the patient's history and lack of tooth mobility accompanied by a clear sound during the percussion examination. To assess the extent of the ankylotic area, RTG or cone-beam computed tomography are used. However, these methods prioritize rapidness over sensitivity, thus preventing any further detailed characterization of this pathology.

Consequently, in this project, a combination of Laser-Induced Breakdown Spectroscopy and Raman spectroscopy is proposed as a way to gain more detailed insight into the composition of the ankylotic pathology. To select a plane in which the LIBS and Raman spectroscopy data will be acquired, micro–Computed Tomography scan of the sample was done. Using the data from the scan, several areas along the tooth root were highlighted as ankylotic. The tooth was then cut to gain a surface with the highlighted ankylotic areas. These areas were then measured by Raman spectroscopy in small-scale maps and consequently, the whole tooth area was measured by LIBS. By analyzing the results from the laser-based methods, the degree of mineralization of the connective soft tissue was determined, and the changes in elemental and molecular composition were quantified and discussed. By fusing the data from both spectroscopies, the level of characterization of the ankylotic area was enhanced. Furthermore, after consulting these results with experts from dentistry, we hypothesize that there could be several stages in the ankylosis development.

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