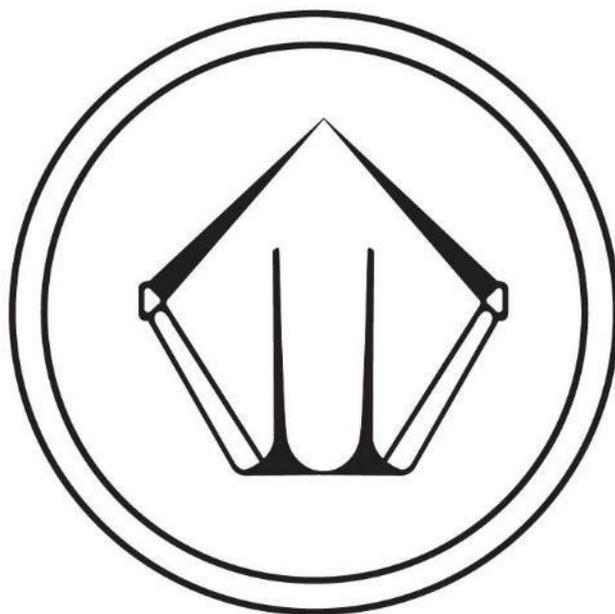


BNASS2020: The 20th Biennial National Atomic Spectroscopy Symposium



28th – 29th June 2022

The Royal Northern College of Music, Manchester



**BNASS is the flagship meeting for the Atomic Spectroscopy Group
of the Analytical Division, Royal Society of Chemistry**



Preface

On behalf of the Organising Committee for the 20th Biennial National Atomic Spectroscopy Symposium (BNASS) and the Atomic Spectroscopy Group (ASG), we would like to welcome you all and thank you for your contributions and participation! A lot has happened since BNASS 2018 but we are very much looking forward to catching up.

The philosophy of BNASS is to encourage the exchange of ideas and knowledge within analytical atomic spectroscopy field with a mix of established and early career speakers. BNASS is celebrating its 20th event and during its history, there have been some tremendous changes in atomic spectroscopy. These advances have been showcased at BNASS, which has built an international reputation for both the quality of the science presented and the unique style of the symposium, enabling delegates to meet and discuss issues in a social setting.

We have found ourselves in Manchester at the Royal Northern College of Music (RNCM) due to Dr Phil Riby who was an integral part of BNASS 2016 in Liverpool and 2018 in Twickenham, as well as the ASG committee. Phil was a reader in Analytical Chemistry at the University of Manchester and was excited to host the next BNASS in Manchester. Very sadly and suddenly, Phil passed away in April 2021 and his lively personality and kind nature are very much missed. He had already played a key role in setting up BNASS for 2020 before the pandemic caused the postponement. Additionally, Phil was a passionate supporter of early career scientists. Therefore, in reflection of this, we are introducing The Phil Riby Award which will be presented to the best student presentation at the conference.

Please enjoy BNASS 2022 and hope that you take full advantage of the networking opportunities with the speakers, attendees and exhibitors. As we look to the future, we hope that BNASS will return to a 3 day format in 2024, alongside a larger celebration. As chair, I would like to thank the organising committee and ASG for their support and dedication.

Organising Committee

ASG Chair: Dr Sarah Hill, National Measurement Laboratory, LGC

Treasurer: Dr Jackie Morton, Biological Monitoring, Health & Safety Executive

Bursary Coordinator: Dr Lizzie Griffin, University of Aberdeen

Programme Book Coordinator: Dr Adam Laycock, UK Health Security Agency

ASG Secretary: Dr Andy Fisher, Plymouth

ASG Committee: Dr Simon Chenery (British Geological Survey), Dr Elliot Hamilton (British Geological Survey), Prof Steve Hill (University of Plymouth)



Venue

The Royal Northern College of Music, 124 Oxford Road , Manchester , M13 9RD, UK
www.rncm.ac.uk

Social Event

On Tuesday evening, we will be hosting a social event at the venue to enable a chance to continue discussions from the day and catch up. It will feature a Manchester speciality - curry! By holding it at the venue, it minimises the number of locations and the venue itself is very airy with plenty of space, seating and air movement.

Wi-Fi

There is Wi-Fi available throughout the venue via the cloud. You can set up an account in advance with your email and create a password <https://www.sky.com/wifi>

Acknowledgements

The Organising Committee wishes to express its grateful thanks to the following organisations for their generous support. BNASS 2022 would not be possible without their involvement and we strongly encourage all attendees to take some time out of the schedule and visit the Vendors Exhibition.

Jump to...

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[Programme](#)

[Invited speakers biographies](#)

Oral presentation abstracts:

- [Session 1](#)
- [Session 2](#)
- [Session 3](#)
- [Session 4](#)

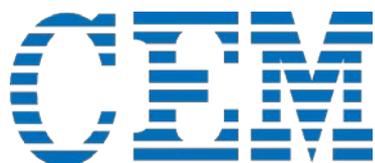
[Poster presentation abstracts](#)



[BNASS Website](#)



SPONSORS



Agilent ICP-QQQ 10-year Birthday Bash!

29 June 2022 | RNCM

Join us in Manchester at RNCM from 2pm

Please join us to celebrate the 10-year anniversary of the introduction of the World's first ICP-MS/MS system – the Agilent 8800 - in 2012.

Learn how the introduction of ICP-QQQ technology has opened up new analytical options for ICP-MS analysis across different applications areas.

Customers who were amongst the first to adopt the 8800 in their labs from 2012 will provide testimonials on how this then revolutionary technology re-shaped the way they work with ICP-MS.

We will share examples from advanced research applications including advances in metrology for speciation, nanoparticle, nuclear and geochemistry applications amongst others and experts from the Agilent R+D team and Applications teams will be at the party to discuss hardware and software developments since 2012 to continually broaden the range of applications for ICP-MS/MS.

We look forward to seeing you at the Birthday Party on June 29th!

With best wishes,

The Agilent Atomic Spectroscopy Team

Please register for the event [here](#)

Event information

Date:

29 June 2022

Location:

Royal Northern College of Music
124 Oxford Rd, Manchester M13
9RD

Time:

14:00 – 17:00



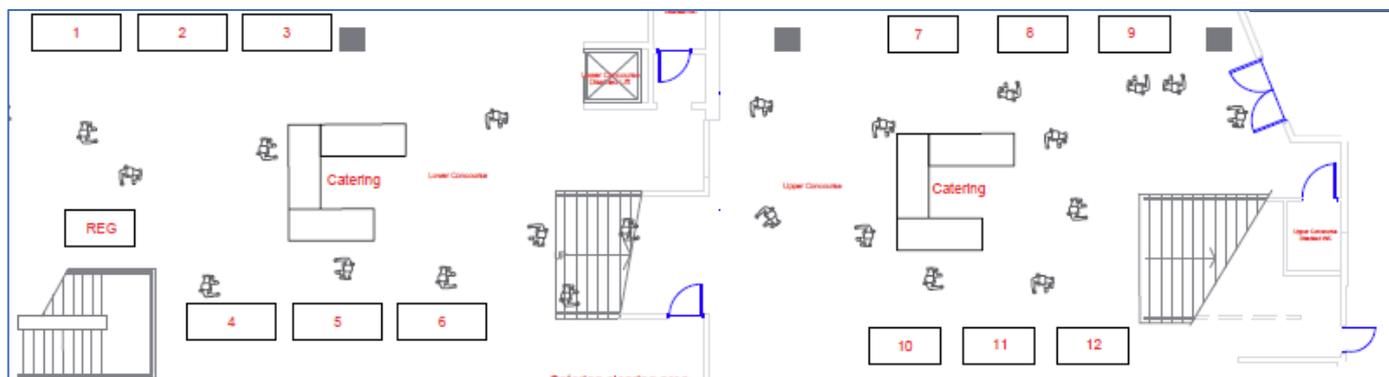
Agilent 8900 Triple Quadrupole ICP-MS System

- Provides configurations offering unrivaled, **consistent** outcomes from routine to research settings
- Cited in over 1000 papers worldwide, including **greener** application areas such as food safety and environmental analysis
- From the earliest Agilent 8800 pioneers in 2012 to our latest Agilent 8900 adopters in 2022, see how ICP-QQQ provides a **smarter** solution for elemental analysis





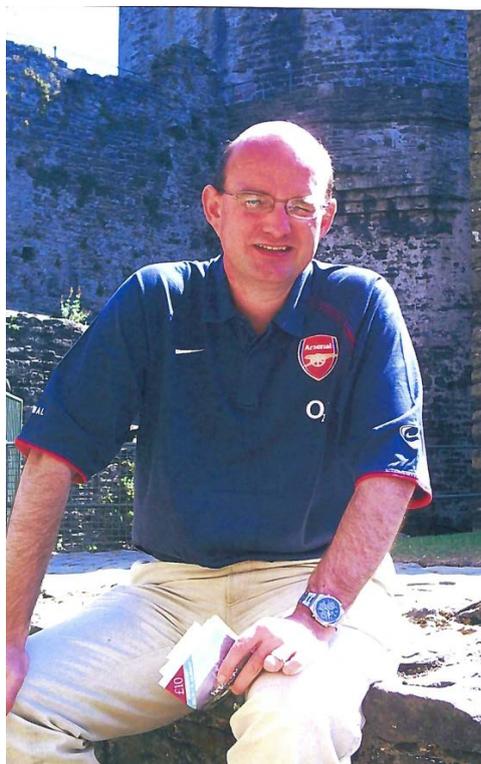
Exhibition Area



Agilent	1
Ametek	2
Analytix	3
Shimadzu	4
QMX	5
SciMed	6
Anton Paar	7
ATG	8
ICPMS Cones	9
Leybold	10
Romil	11
CEM	12



In Memory of Dr Phil Riby



It was with great sadness that the BNASS committee learnt of the untimely passing of Phil Riby in April 2021. Phil was Chair of the 18th BNASS meeting in Liverpool, on the organising committee of 19th BNASS in London, and was instrumental in bringing BNASS to Manchester this year. His enthusiasm, drive and cheerful nature will be greatly missed. Phil was Reader in Analytical Chemistry at the University of Manchester for nearly 5 years. Prior to that he spent over 14 years at Liverpool John Moores University, a post he took after 11 years at the University of Greenwich. Phil also undertook his PhD at Greenwich and returned there after 2 year post-doctoral experience at the United States Department of Agriculture. He obtained his first degree from Sheffield Hallam University graduating in 1986. Phil's main research area was atomic spectroscopy and microwave sample preparation, although he did migrate into chromatography whilst at Manchester. However, above all Phil was a people person, and in particular, a champion for younger scientists. The welfare and success of his students was his priority at all times. He was also devoted to his family, his wife Paula and two daughters Sorcha and Niamh. In order to commemorate Phil's contribution to BNASS, education and the wider atomic spectroscopy community, this year the BNASS committee will be awarding a student prize in his memory. The Phil Riby Award will be presented for the best student presentation at the conference.



Professor Stanley Greenfield CChem FRSC:

25 March 1920 – 4 September 2019.



Delegates are invited (by visiting poster 6A&B) to take some time to acknowledge and reflect on the contributions by [Stanley Greenfield](#) (pictured here in 2004) to the development of plasma spectrochemistry and to the Analytical Division of the Royal Society of Chemistry. Instrumentation featuring ICP sources, for



both emission and mass spectrometries, is now so widely used in laboratories around the world that it is easy to overlook that this was not always the case. Not only was Stan a visionary scientist, he was also dedicated to the success of the AD/RSC in whose operations he was intimately involved from the time he joined the Society for Analytical Chemistry in 1955 (at age 35) to around 2015 (at age 95)—an astonishing 60 years of service.

Stan was unusual in many respects, but crucial to his successful demonstrations of the capabilities of the ICP as a source for atomic emission spectrometry was his employment. Unlike all other researchers whose names appear in the [early history of ICP-AES/MS](#), he worked for a chemical company, not at a university or for an analytical instrument company. In the early 1960's, multi-element determinations were a significant challenge. The only options available were arc/spark and flame emission spectrometries, both of which had serious limitations for the analyses of the various non-metallic samples encountered in Albright and Wilson's phosphorus development department: contamination by electrode materials, matrix interferences, and inadequate detection limits.

Stan's research group realized that the way forward was to stimulate atomic emission from a source (a) whose temperature was sufficiently high to prevent stable compound formation, and (b) that could accept fluid samples (either acid solutions or powders). They had the resources to assemble three candidates: two types of DC arc plasma-jet and an ICP. In 1964, in what is [considered](#) a landmark publication,¹ they showed the very considerable advances in analytical capability provided by the ICP source. A crucial feature of their plasma, not appreciated or achieved by contemporaneous researchers, was that it was *annular*, allowing sample aerosol to flow through a central channel into a spectroscopically useful "tail-flame." Some years later they reported² on extensive successful practical applications. And the rest, as they say, is history. Right up until his retirement from A&W in 1980, Stan was an enthusiastic advocate for the ICP: he lectured extensively both in the UK and around the globe and maintained a steady output of both research papers and authoritative reviews.

The remaining problem of spectral interferences in AES could, he argued, be overcome by switching to atomic fluorescence spectrometry (AFS) and for the next 10



years or so, he investigated (first at [Loughborough](#) and then at Plymouth Universities) the analytical possibilities of AF stimulated by radiation from a (high-power) ICP (or hollow cathode lamp) in atoms generated in another (low-power) ICP. He presented a progress report in an invited lecture at the 1994 BNASS meeting in Hull.³ His last publication⁴ was in 2003 (at age 83).

Stan's contributions to the [AD/RSC](#) were, to say the least, extensive. He served for many years on the Council, including as [President of the Division](#) (1982-1984), he was active in the running of (a) the Midlands Region, (b) the Special Techniques Group, (c) many SAC conferences, and perhaps most extraordinary of all, as a member of the [Analytical Methods Committee](#) whereby he oversaw, as founding chair of the [Instrument Criteria sub-Committee](#), the creation and publication of some 27 reports over a 21 year-period that only ended in 2015 (at age 95).

1. S. Greenfield, I. Ll. Jones and C. T. Berry, [High-pressure plasmas as spectroscopic emission sources](#), *The Analyst*, 1964, **89**, 713.
2. S. Greenfield, I. Ll. Jones, H. McD. McGeachin and P. B. Smith, [Automatic multi-sample simultaneous multi-element analysis with a h.f. plasma torch and direct reading spectrometer](#), *Anal. Chim. Acta*, 1975, **74**, 225–245.
3. S. Greenfield, [Inductively coupled plasma in fluorescence spectrometry: source and atom/ion reservoir. Invited lecture](#), *J. Anal. At. Spectrom.*, 1995, **10**, 183.
4. A. Young, L. Pitts, S. Greenfield and M. Foulkes, [A preliminary comparison of radial and axial excitation fluorescence in the ICP using non-laser sources](#), *J. Anal. At. Spectrom.*, 2003, **18**, 44–48.



Time	Tuesday 28 th June: Main Programme
9.00-9.30	Registration
9.30-9.45	Welcome address: BNASS 2022 Organising Committee
Session 1: Clinical, Biomedical and Health Applications Chair: Dr Jackie Morton	
9.45-10.15	Keynote 1: Maria Montes-Bayón, University of Oviedo, Oviedo, Spain <i>Quantitative Biomarker Analysis Via ICP-MS: A Focus On The Breast Cancer Marker (HER-2)</i>
10.15-10.35	Joshua Millar, Sheffield Hallam University, Sheffield, UK <i>Multimodal mass spectrometry imaging of key species to study age-related macular degeneration</i>
10.35-10.55	Lukas Schlatt, Nu Instruments, Wrexham, UK Endogenous metal analysis in labelled single cells using time of flight ICP-MS for the elucidation of a cells metalome on a per cell level
10.55-11.30	Refreshment break
11.30-11.50	Chris Harrington, Trace Element Laboratory, Royal Surrey Hospital Copper Measurements in Wilson Disease
11.50-12.10	Aristea Anna Leventi, University of Strathclyde, Glasgow & NML at LGC, Teddington, UK Potential Of Quantitative LA-ICP-TOF-MS To Underpin Absolute SERS Quantitation In A Cancer Model
12.10-12.45	Lightning poster talks
12.45-13.45	Lunch
Session 2: Speciation and Environmental Chair: Dr Simon Chenery	
13.45-14.15	Keynote 2: Andrea Raab, TESLA (trace element speciation laboratory) University of Graz, Austria Elemental speciation – essential or only play?
14.15-14.35	Rebecca Sim, Matis, Reykjavík, and University of Iceland, Reykjavík, Iceland Do sample preparation methods change the distribution of water-soluble arsenicals in <i>Laminaria digitata</i> ?
14.35-14.55	Joseph Ready, Sheffield Hallam University, Sheffield, UK The Use of LA-ICP-MS and Related Techniques for the Analysis of Essential Elements in Plant Tissue
14.55-15.15	Neelam Manzoor, University of Strathclyde, Glasgow, UK Influence of washing reagent used on concentration of potentially toxic elements determined in bioindicator plants
15.15-15.45	Refreshment break
15.45-16.15	Keynote 3: Dr David Green, Imperial College London, London, UK Atmospheric Particulate Measurement and Source Apportionment using High Time Resolution X-ray Fluorescence and Aerosol Mass Spectrometry
16.15-16.35	Rebecca Vesuwe, University of Strathclyde, Glasgow, UK Occurrence and oral bioaccessibility of potentially toxic elements in crude oil contaminated soils.
16:35-16.55	Elnaz Barati Imperial College London, London, UK Investigating Cadmium and Zinc Interactions in Cacao Using Stable Isotope Analyses
16.55-17.25	Lightning poster talks
17.25-18.30	Drinks reception and poster session
18.30-19.00	Manufacturers in the “Atomic Spectroscopy Dragon’s Den” Instrument manufacturers face the Atomic Spectrometry Panel
19.00	Social Evening Event, ‘Curry buffet and local drinks’.



Time	Wednesday 29th June: Main Programme	
Session 3: Advances in Measurement Applications		Chair: Dr Sarah Hill
09.30-10.00	Keynote 4: Heidi Goenaga-Infante, NML at LGC, Teddington, UK Nanomaterials in life sciences: Advances in Standardization, Measurement Methods, Reference Materials and Remaining Challenges Imposed by Regulation	
10.00-10.20	Simon Chenery British Geological Survey, Nottingham, UK Adventures in Elemental Mapping with Laser Ablation – ICP-MS	
10.20-10.40	Shaun Lancaster, Montanuniversität Leoben, Leoben, Austria Exploring the use of nitrous oxide as a cell gas for inductively coupled plasma tandem mass spectrometry measurements	
10.40-11.00	Ben Russell, National Physical Laboratory, Teddington, UK Comparison of tandem inductively coupled plasma mass spectrometry (ICP-MS/MS) for nuclear and gas particle metrology applications	
11.00-11.30	Refreshment break	
Session 4: Instrumental Advances		Chair: Dr Heidi Goenaga-Infante
11.30-11.50	Matthew Duggan, University of Manchester, Manchester, UK Identification of ⁹⁰ Sr in environmental samples via the hyphenation of ICP-MS with Collinear Resonance Ionisation Spectroscopy	
11.50-12.10	David Price, Perkin Elmer, Seer Green UK Work those quads-going the distance with Multi Quad ICP-MS	
12.10-12.30	Rai Wahlen, Agilent, Stockport, UK 10 years of ICP-MS/MS: How ICP-QQQ has changed ICP-MS applications and markets	
12.30-12.50	David Bellis, SciMed, Stockport, UK How many Quads do you need? Choosing the right tool for right job.	
12.50-13.00	Close and Final Remarks: BNASS Organising Committee	
13.00	Lunch and depart	
14.00-17.00	Agilent ICP-QQQ 10-year Birthday Bash!	



Keynote Speakers

Keynote Presentation 1, Tuesday 09.45

Quantitative Biomarker Analysis Via ICP-MS: A Focus On The Breast Cancer Marker (HER-2)

Maria Montes-Bayón, University of Oviedo, Oviedo, Spain



Maria Montes-Bayón is Full Professor in Analytical Chemistry in the Department of Physical and Analytical Chemistry at the University of Oviedo (Spain) since December 2017.

Prof. Maria Montes-Bayón holds a bachelor in Chemistry (1993, University of Oviedo, Spain), bachelor-thesis in Analytical Chemistry (1994, University of Plymouth, UK and Oviedo) and a PhD (1999, University of Oviedo; awarded) degree. In the year 2000, she joined the research group of Prof. Joseph A. Caruso at the Department of Chemistry at the University of Cincinnati (Ohio, USA) as Postdoctoral Fulbright Fellow. She got back to Oviedo with a Senior Researcher contract and was promoted in 2008 to Associate Professor and in 2017 to Full Professor of Analytical Chemistry. Her main research interests are: a) Development of quantitative strategies for determination of molecular biomarkers of clinical relevance at the single cell level using ICP-MS; b) Evaluation of encapsulated metallodrugs for their potential use in chemotherapy using mass spectrometry: a focus on cellular resistance mechanisms; and c) analytical tools to study biomedical aspects of nanoparticles: biogenic nanoparticles and metal nano-debris from metallic implants.

She has supervised 14 PhD students since 2007 and mentor of 5 postdocs who built up a successful career afterwards. She has given >40 invited and plenary lectures in national and international congresses and conferences and co-authors 164 publications (80% in Q1; 32 with >50 citations), including original research articles, reviews, and several book chapters (with >5,300 total citations, H-index=42). She has got funding through 29 international/national/regional research projects (12 of the as PI) and is associate editor of the journal *Metallomics* (RSC/OUP) since 2015. Currently, she has been elected as President of Spanish Society for Analytical Chemistry since 2022.



Keynote Presentation 2, Tuesday 13.45

Elemental speciation – essential or only play?

Andrea Raab, TESLA (trace element speciation laboratory) University of Graz, Austria



Andrea Raab started working in metalloprotein speciation at the Hahn-Meitner-Institute Berlin using size-exclusion chromatography coupled to ICP-OES and ICP-MS. On joining TESLA in Aberdeen her work changed to small molecule speciation especially arsenic-containing compounds with different separation methods. Her main interests now are parallel application of elemental and molecular mass spectrometry for small hetero-atom containing compounds, proteins and peptides, beside quantitative bio-imaging of tissues. Today she manages the TESLA laboratories beside being active in research.



Keynote Presentation 3, Tuesday 15.45

Atmospheric Particulate Measurement and Source Apportionment using High Time Resolution X-ray Fluorescence and Aerosol Mass Spectrometry

David Green, Imperial College London, London, UK



Dr David Green is a Senior Research Fellow in the Environmental Research Group where he leads the Aerosol Science Team. Research interests focus on the measurement of particles and gases, and their use in source apportionment and health studies, to improve our understanding in both the urban and global environment.

Measurement forms a key part of the research and includes a significant suite of analytical equipment deployed either in the laboratory or in the field. This is principally focused in 'supersites' in London, monitoring and disseminating the chemical and physical composition of aerosols in real time to inform forecasting and public information systems.

The measurements are also used in epidemiological studies to assess the impact of particles and their different composition on health end points.

Source apportionment methodologies are used to interpret the high time resolution chemical composition measurements and quantify the contribution of different emission sources in complex urban environments. In these environments a wide variety of sources, both local (such as traffic) and distant (such as that from agricultural sources), contribute to the total particle load in the atmosphere.



Keynote Presentation 4, Wednesday 9.30

Nanomaterials in life sciences: Advances in Standardization, Measurement Methods, Reference Materials and Remaining Challenges Imposed by Regulation

Heidi Goenaga-Infante, NML at LGC, Teddington, UK



Heidi's scientific career of over twenty years of experience on elemental and speciation analysis started with the award of a PhD from Oviedo University, Spain. She joined LGC as senior researcher in speciation analysis in 2003. Currently, she is a Science Fellow of LGC. She is also the Principal Scientist and Team Leader of the Inorganic Analysis team of sixteenth PhDs and postgraduate scientists. Her group expertise currently lies in trace element speciation analysis, metallomics research, the characterisation of nanomaterials, high accuracy isotope ratio

analysis, quantitative elemental bio-imaging and the characterisation of "speciated" reference materials and standards. Heidi Goenaga-Infante is the UK representative at the Inorganic Analysis Working Group of the CCQM, the international Consultative Committee for Metrology in Chemistry. She is chair of the Editorial Board of the Journal of Analytical Atomic Spectrometry and, a member of the international advisory board of Analytical and Bioanalytical Chemistry. She is the Government Chemist representative on the Nanomaterials Environment and Health Government Group chaired by DEFRA and the LGC representative at ISO TC 24 (Particle characterisation). She is the EURAMET representative for inorganic analysis at the CCQM Key Comparison Working Group and a member of IUPAC. She has acted as the coordinator of the EU EUROPEAN Metrology Research Proposal (EMRP) NanoChop "Chemical, Optical and Biological characterisation of nanomaterials in biological samples". She is the leading author of over a 140 scientific research papers and 8 book chapters. Heidi was awarded the 2020 Lester W. Strock Award from SAS.



Session 1: Clinical, Biomedical and Health Applications

Tuesday 28th 9:45

Quantitative Biomarker Analysis Via ICP-MS: A Focus On The Breast Cancer Marker (HER-2)

Maria Montes-Bayon^{1,2}, Alejandro Fernandez-Asensio¹, Mario Corte-Rodriguez^{2,1}, Luisa Maria Sierra Zapico^{1,2}, Elisa Blanco González^{1,2}

¹University of Oviedo, Oviedo, Spain.

²Instituto de Investigación Sanitaria Principado de Asturias (ISPA), Oviedo, Spain

Breast cancer is a disease with high incidence, high mortality rate, and high economic and social costs. Clinically, breast cancers are categorized into one of three major subtypes, using the three standard immunohistochemical markers: estrogen receptor (ER), progesterone receptor (PR) and human epidermal growth factor receptor 2 (HER-2) being the HER2-positive an aggressive type of breast cancer and tend to grow faster and are more likely to spread. Thus, HER-2 protein acts as an important biomarker and target of therapy for about 30% of breast cancer patients. The early detection of HER-2 positive tumor cells is, therefore, crucial for the future outcome of the affected patients.

In this work, we present two different strategies based on ICP-MS detection to detect HER-2 in cell cultures. Both methods are centered on immunological reactions with different reporting molecules. In one of them, the so called immune-PCR, combines the selectivity of the immunochemical reaction, the amplification by PCR of the reporting molecule (a custom oligonucleotide) and the detection of phosphorous in the amplicon by means of triple quadrupole ICP-MS. Another strategy, will based on the labelling of individual cells using antibodies containing metal probes in combination with ICP-MS fitted with single cell sample introduction systems. The evaluation of the assay in mixtures containing HER-2 positive and negative will be highlighted.



Session 1: Clinical, Biomedical and Health Applications

Tuesday 28th 10.15

Multimodal mass spectrometry imaging of key species to study age-related macular degeneration

Joshua Millar¹, Susan Campbell¹, Catherine Duckett¹, Sarah Doyle², Laura Cole¹

¹Sheffield Hallam University, Sheffield, United Kingdom.

²Trinity College Dublin, Dublin, Ireland

Essential and non-essential trace metals alongside proteins associated with the innate immune system are linked with age-related macular degeneration (AMD). Determining the distribution of metals within ocular tissue, and mapping metal accumulation in the elderly is important in understanding connections between dysregulation and onset of AMD. Additionally, determining the location of innate immune receptors thought to be involved in disease pathogenesis is important when assessing how best to target these receptors. Here, metallomic analysis was conducted on mouse ocular tissue using ImageBio266 and UP-213 laser ablation (LA) units (Elemental Scientific Lasers, Bozeman, CA, USA) operating at 10 μ m and 6 μ m respectively coupled to a NexION 350x ICP-MS (Perkin Elmer, Waltham, MA, USA) proteomic data was acquired using a SYNAPT G2 MALDI-MS (Waters Corporation, Manchester, UK). LA-ICP-MS has been performed to characterise accumulation of both essential and non-essential metals within mouse ocular tissues, high resolution mass spectrometry aimed to determine spatial distribution of key trace metals within the mouse chorio-retinal microanatomy. Moreover, MALDI-MS has exhibited the distribution of NLRP3 and associated proteins in mouse retinal tissue. This study exhibits the capabilities of multimodal MSI, and how ICP-MSI and MALDI-MSI have potential to inform on novel biological mechanisms of retinal degeneration.



Session 1: Clinical, Biomedical and Health Applications

Tuesday 28th 10.35

Endogenous metal analysis in labelled single cells using time of flight ICP-MS for the elucidation of a cells metalome on a per cell level

Lukas Schlatt¹, Phil Shaw¹

¹Nu Instruments, Wrexham, United Kingdom

Mass cytometry is a valuable tool for investigating protein expression by individual cells within heterogeneous cellular suspensions. With this approach, heavy metal isotope conjugated probes – typically antibodies – label multiple cellular proteins, and TOF-ICP-MS is then used to simultaneously detect these probes on single cells. Analysis of the resulting data allows population heterogeneity to be determined at the individual cell level.

In many applications it is crucial to not only detect the metal labels, which allow the differentiation of cells, but also endogenous elements. When combining the information from all elements, the metalome can be examined on individual cell level. Herein, data recorded on a Vitesse TOF-ICP-MS is shown. The high acquisition speeds allow for precise cell identification and integration in the NuQuant software, while the full elemental capabilities enable the detailed examination of the metalome of single cells. Data reduction methods were possible using both Python scripts within NuQuant as well as output in Flow Cytometry Standard (FCS) format, which is compatible with commonly used flow cytometry data analysis pipelines.



Session 1: Clinical, Biomedical and Health Applications

Tuesday 28th 11:30

Copper Measurements in Wilson Disease

C.F. Harrington¹, A. Ala, G. Carpenter¹, K. Willis¹, M. Schilsky¹

¹Supra-Regional Assay Service, Trace Element Laboratory, Surrey Research Park, Royal Surrey Hospital, Egerton Road, Guildford, UK

Wilson disease, first described by Kinneer Wilson in 1912 is a rare autosomal recessive genetic disorder which affects copper metabolism and has an estimated prevalence between 1:30,000 and 1:50,000. Intracellular copper is normally incorporated into ceruloplasmin in hepatocytes, a secretory glycoprotein that incorporates copper during its biosynthesis, aided by the P-type ATPase copper transporter protein *atp7b*. The build-up of intracellular copper that is due to failure of the transfer of excess hepatocellular copper into bile, eventually leads to copper accumulation, first in the liver, then throughout the body, resulting in copper-induced liver injury and eventual cirrhosis and neurological complications. Untreated the condition is fatal.

The diagnosis of Wilsons disease, involves the pathological investigation of patient samples. Classically, this has involved the measurement of the copper (Cu) content of serum or plasma, urine Cu excretion and Cu in liver biopsies. The determination of the Cu containing protein ceruloplasmin has also often been measured in serum or plasma to aid in the diagnosis but also to calculate the so call “non-ceruloplasmin bound Cu” (NCC). However, because of the impact of nutritional intake, malabsorption and other diseases, particularly other liver disease, these indices do not always translate into a clear cut and easily interpretable diagnosis. It is for this reason that other biomarkers for Wilson disease have been sought including, the measurement of “free” or exchangeable Cu and the relative exchangeable Cu.

This talk will explain these clinical tests, how the newer biomarkers can be used and their advantages and drawbacks in diagnosis and monitoring. It will then go on to discuss the use of more advanced protocols that can be used to elucidate the metabolism of Cu and Cu-containing proteins. These methods developed in the authors laboratory, use strong anion exchange chromatography (SAX) coupled to triple quadrupole inductively coupled plasma mass spectrometry (HPLC-ICPMS/ MS) to overcome the drawbacks of methods for the measurement of all copper-containing metalloproteins in serum.

Using the developed system we have shown that the approach is a useful addition to the methods available for the investigation of Cu metabolism in human studies and in particular patients with Wilsons disease.



Session 1: Clinical, Biomedical and Health Applications

Tuesday 28th 11:50

Potential of Quantitative LA-ICP-TOF-MS to Underpin Absolute SERS Quantitation in a Cancer Model

Aristea Anna Leventi^{1,2}, Kharmen Billimoria², Dorota Bartczak², Gonzalo Huelga-Suarez², Heidi Goenaga-Infante², Karen Faulds¹, Duncan Graham¹

¹University of Strathclyde, Glasgow, United Kingdom.

²NML at LGC, Teddington, United Kingdom

Elucidation of the underlining mechanisms of cancer by detecting and quantifying key biomarkers via tagged antibodies at a single cell level is an area of increasing interest and requires validated multi-modal analytical platforms to quantify their intra-cellular distribution. Surface enhanced Raman spectroscopy (SERS), involving nanomaterials, has been widely explored as the non-destructive technique of choice for this purpose. However, the capability of absolute quantitation remains a challenge for this technique.

This poster will discuss development of a multi-modal platform involving particle counting and fast LA-ICP-ToF-MS imaging strategies for the characterisation of suitable SERS nanoprobe consisting of gold nanoparticles (AuNPs) and the SERS reporter 1,2-bis(4-pyridyl)ethylene (BPE). Results from extinction spectroscopy and particle tracking analysis showed a monomodal character of the SERS probes (diameter of approximately 36.7 nm) and demonstrated their colloidal stability for 2 months. Moreover the relationship between BPE and Au was established using SERS and single particle ICPMS. Calibration standards were prepared by 3D printing of gelatin droplets spiked with Au:BPE nanoprobe. Analysis of the calibration standards by LA-ICP-ToF-MS at a spatial resolution of 5 μm , demonstrated the homogeneous distribution of Au:BPE nanoparticles in the droplets and the linearity of ¹⁹⁷Au response with increasing Au concentration in the standards (R^2 : 0.9929). The standards were analysed by SERS mapping and different data processing approaches were investigated. The resulting calibration model was based on the BPE signal, correlating the SERS response to the total Au concentration (R^2 : 0.9029), demonstrating their potential for further use for SERS calibration.



Session 2: Speciation and Environmental

Tuesday 28th 13.45

Keynote: Elemental speciation – essential or only play?

Andrea Raab¹

¹TESLA (trace element speciation laboratory Graz), University of Graz, Graz, Austria

Elemental speciation is defined as “the detection and quantification of the different molecular species of an element present in a given sample”. What is considered a species is operationally defined, since electrochemists define species potentially differently than someone using HPLC-ICPMS or HPLC-ESMS. Here the focus will be on species as separable by HPLC and detectable by mass spectrometers.

One of the often-posed questions regarding elemental speciation is: “Is it worth the effort?”

Elemental speciation has since its beginnings made great progress and is now more routinely used, but still not commonplace. One of the reasons for this is that the detector used mostly is an ICPMS which is relative expensive to run. In addition, the software controlling the instruments is not yet that perfect to allow simultaneous control of an HPLC and the data analysis still leaves something to desire. So, when and why go the additional step and do speciation instead of simple elemental quantification?

Elemental speciation can be advisable when:

- Different elemental species show different toxicity (e.g. arsenic)
- It is necessary for the unravelling of the elemental metabolism, especially when the element in question forms stable element-carbon bonds (e.g. arsenic / selenium)
- When different elemental species show different bio-accumulation potential (e.g. mercury)
- Identification of unknown compounds (e.g. fluorine)



Session 2: Speciation and Environmental

Tuesday 28th 14.15

Do sample preparation methods change the distribution of water-soluble arsenicals in *Laminaria digitata*?

Rebecca Sim^{1,2}, Jörg Feldmann³, Ásta Pétursdóttir¹

¹Matís, Reykjavík, Iceland.

²University of Iceland, Reykjavík, Iceland.

³University of Graz, Graz, Austria

Sample pre-treatment is perhaps the most critical step in arsenic speciation. Typically drying methods involving heat or freezing are used to prevent unwanted chemical or microbial changes to samples. Few studies report results obtained from extractions using fresh seaweed, and so little is known about whether the integrity of the arsenic species – in particular arsenosugars – is maintained during the drying procedure.

The aim of this study was to determine whether the drying techniques commonly used to preserve biological samples change the distribution of water-soluble arsenicals in seaweed. The anatomical sections as well as whole thalli were analysed in an effort to provide insight into how and where these arsenosugar species are formed within the seaweed. *Laminaria digitata* individuals were collected from a beach near Grindavík, Iceland in 2022. The whole thalli samples were homogenised and dried in one of three ways – freeze-dried, air-dried or oven dried – and a fourth batch underwent no drying and immediate extraction with water. The anatomical sections of *L. digitata* were subjected to either freeze-drying or no drying.

The inorganic arsenic was consistently found to increase along the thallus from stipe to distal frond, and the dominant arsenosugar differed depending on section of the thallus. The results of this study are discussed further in the contents of the poster.



Session 2: Speciation and Environmental

Tuesday 28th 14.35

The Use of LA-ICP-MS and Related Techniques for the Analysis of Essential Elements in Plant Tissue

Joseph Ready¹, Kathryn Knight², Natasha Hall², Laura Weatherhead², Martin Shaw², Laura Bishop³, Andy Fowles³, Catherine Duckett¹, Malcolm Clench¹, Neil Bricklebank¹

¹Sheffield Hallam University, Sheffield, United Kingdom.

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Metals are essential for normal plant development and are involved in growth, regulation and metabolism. Maintaining the nutritional metal content in plants is crucial. The work aimed to use LA-ICP-MS bioimaging alongside complementary techniques to determine the effect of the inclusion of adjuvants in a zinc-containing seed film coating, on the uptake of Zn into wheat crops.

Laser Ablation was performed using an Elemental Scientific Lasers imageBIO266 Laser Ablation System, coupled to a Perkin Elmer Nexlon 350X ICP-MS.

Analyses were carried out on treated seeds over 12 months to investigate the impact of the inclusion of adjuvant on the stability of the seed film coating after storage, and to investigate the mechanisms by which seed-applied zinc was uptake by wheat seeds with and without the presence of adjuvant in the film coating formulation. Imaging studies carried out at 12 months post-treatment suggest that the inclusion of adjuvant may increase the uptake of Zn during the imbibition process of wheat seeds. This is supported by bulk analysis data, along with imaging studies carried out using micro-computed tomography (micro-CT).



Session 2: Speciation and Environmental

Tuesday 28th 14.55

Influence of washing reagent used on concentration of potentially toxic elements determined in bioindicator plants

Neelam Manzoor¹, Christine M. Davidson¹

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Exposure to potentially toxic elements (PTEs) associated with airborne particulate matter (APM) can result in serious human health problems. Therefore, determination of PTE concentrations in APM is important. Various kinds of plants have been used as bio-monitors for airborne PTE, where the analyte levels associated with deposited APM are estimated by comparison of results obtained for washed and unwashed plant material. In this study four reagents – distilled water, HCl, Tween 80, and Na₂EDTA – were used to wash plants of different morphologies (moss, dandelion, rhododendron and thuja). Samples were then ground and subjected to microwave-assisted digestion followed by ICP-MS analysis for Cd, Cr, Cu, Mn, Pb and Zn. The HCl removed the largest amounts of: Cd (76.7%), Cu (15%), Mn (35.2%), Pb (41.8%) and Zn (52%) in moss; Mn (48.4%) in dandelion; Cu (25.1%), Mn (24.9%), Pb (52.5%) and Zn (29.9%) in rhododendron; and Cu (13.1%), Mn (59.4%), and Pb (53%) in thuja. The largest amounts of Cr were removed from moss (43.5%), dandelion (87.3%), and thuja (27%) when Tween 80 was used. Tween 80 was also most effective in removing Cu (22.3%), Pb (66.9%) and Zn (70.4%) from dandelions and Cr (27%) from thuja. Distilled water and Tween were equally effective in removing Pb (52.4%) from rhododendrons, whilst distilled water, Na₂EDTA and Tween all removed around 57% of Cr from rhododendron. Overall, HCl proved to be most effective washing reagent, except for Cr where Tween 80 was most effective. Washing samples with Tween 80 followed by HCl is therefore recommended.



Session 2: Speciation and Environmental

Tuesday 28th 15.45

Keynote: Atmospheric Particulate Measurement and Source Apportionment using High Time Resolution XRF and Aerosol Mass Spectrometry

David Green¹

¹Imperial College London, London, UK

Airborne particulate matter (PM₁₀ and PM_{2.5}) has long-established health and climate forcing impacts. Recent advances in the field deployable mass and x-ray spectroscopy instrumentation have enabled a more detailed analysis of the chemical composition of these aerosols at higher time resolution. This has resulted in a greater understanding of the contribution of different sources and atmospheric processes that lead to poor air quality and is of fundamental importance to the environment and public health. The use of these measurement techniques in national and international measurement campaigns and networks will be explored, particular emphasis will be placed on the advances in understanding of the current sources of PM₁₀ and PM_{2.5} in urban environments.



Session 2: Speciation and Environmental

Tuesday 28th 16.15

Occurrence and oral bioaccessibility of potentially toxic elements in crude oil contaminated soils.

Rebecca Vesuwe¹, Christine Davidson¹

¹University of Strathclyde, Glasgow, United Kingdom

Spills of crude oil on soil leads to contamination of the soil with potentially toxic elements (PTEs), which persist in the soil for a long time, bioaccumulate and cause negative effects on human health and the environment. There is therefore a need to better understand the levels and distribution of crude-oil derived PTEs in the environment, and levels available for absorption into the body.

In this study, the pseudototal and oral bioaccessible concentrations of As, Cd, Cr, Cu, Ni, Pb, V and Zn were determined using ICP-MS. Soil samples were collected from five communities in Gokana Local Government Area of Rivers State, Nigeria. Samples were microwave digested using a mixture of HCl and HNO₃ in the ratio 1:3 (reverse aqua regia), following a 4 hours pre-digestion time. Oral bioaccessibility test was performed on samples that had higher PTE concentration, using the SBET method.

The pseudototal concentrations of the PTEs from the wet, dry and bunkery soils ranged from 0.39 – 4.67 mg/kg As; <0.01- 0.17 mg/kg Cd; 1.65 – 99.2 mg/kg Cr; 1.83 – 216 mg/kg Cu; <0.01 – 14.9 mg/kg Ni; 1.13 – 447 mg/kg Pb; 1.99 – 27.9 mg/kg V and 2.88 – 549 mg/kg Zn. While bioaccessible concentrations ranged from 0.86 – 3.28mg/kg As; <0.01 – 0.28 mg/kg Cd; <0.01 – 6.56 mg/kg Cr; <0.01 – 119 mg/kg Cu; < 0.01 – 8.42 mg/kg Ni; <0.01 – 243 mg/kg Pb; 0.39 – 5.49 mg/kg V and 0.79 – 2.31 mg/kg Zn. Concentrations of PTEs at some sample points during the dry season were above maximum permissible limits for soils.



Session 2: Speciation and Environmental

Tuesday 28th 16.35

Investigating Cadmium and Zinc Interactions in Cacao Using Stable Isotope Analyses

Elnaz Barati¹, Rebekah Moore¹, Ihsan Ullah², Katharina Kreissig¹, Barry Coles¹, Jim Dunwell², Mark Rehkämper¹

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²University of Reading, Reading, United Kingdom

To help address the growing health concerns associated with the presence of cadmium (Cd) in cocoa-based products, the EU established new strict upper limits for Cd present in cocoa powder sold to consumers. Research is being undertaken to understand the underlying origins of and/or mechanisms for the observed Cd enrichments in the beans of *Theobroma cacao*.

As zinc (Zn) and Cd have similar chemical and physical properties, the two elements are expected to undergo similar biological and physicochemical processes in plants during uptake, storage, and translocation to aerial plant parts. By understanding if Zn and Cd use similar mechanisms to enter and move around in plants, agricultural and land management practices can be implemented to reduce the uptake and translocation of Cd in cacao plants.

Using anion exchange chromatography and multiple collector ICP-MS, Zn stable isotope and concentration analyses were conducted on juvenile seedlings from 19 different genetically diverse genotypes of cacao. Cadmium was also added to the hydroponic nutrient solutions. The results show all genotypes preferentially take up isotopically light Zn. Furthermore, the roots are enriched in isotopically heavy Zn isotopes, whilst isotopically light Zn is preferentially transferred to aerial plant parts. This isotope partition trend between roots and aerial plant parts is opposite to what was observed for previously published Cd stable isotope data from the same plants. As with the findings from Zn, cacao plants preferentially take up isotopically light Cd. This study contributes to understanding how agricultural practices can produce cacao beans with limited Cd.



Session 3: Advances in Measurement Applications

Wednesday 29th 09.30

Keynote: Nanomaterials in life science applications: The role of reference methods and reference materials to support upcoming regulation

Heidi Goenaga Infante¹, Isabel Abad-Alvaro¹, Susana Cuello-Nunez¹, Dorota Bartczak¹

¹LGC Limited, Queens Road, Teddington, Middlesex TW11 0LY, United Kingdom

The EU definition of nanomaterials (2011/696/EU) as well as existing regulations (e.g. Cosmetics 1223/2009, Novel food 2015/2283, Medical Device 2017/745) have posed several analytical challenges as well as driven new measurement strategies and standards in the past ten years. Although key analytical advances have been reported recently, there is still an increasing need for traceable methods and reference materials for quality assurance of nanoparticle characterisation in complex matrices. Such materials and methods will help support upcoming regulation and enable quality control of existing products.

This lecture will cover in particular recent guidance and scientific opinions on the safe use of nanomaterials and key regulations, which have emerged from the increasing use of nanomaterials in several industrial applications. It will also discuss the measurement capability needed to comply with those regulations and for future risk assessment. It will also touch on measurement lessons learnt from international metrology intercomparisons, highlight future measurement and technology requirements and associated challenges and, provide information on the existing reference materials for method development and validation with emphasis on remaining gaps for matrix/analyte combinations.

Finally, the lecture will discuss existing analytical technology and advances in measurement capabilities for the characterization of nanomaterials and their input on key relevant ISO standards. Focus will be on inorganic nanomaterials and on multi-modal platforms combining hyphenated ICP-MS with spectroscopy and microscopy techniques. Examples of key analytical challenges and recommendations for overcoming such challenges will be given through the characterization of complex nanomaterial mixtures released from medical devices [1-3] by single particle ICP-MS and AF4-ICPMS/light scattering for toxicology testing.

[1] ISO/TR 10993-22:2017 - Biological evaluation of medical devices — Part 22: Guidance on nanomaterials

[2] ISO 10993-1:2018 - Biological evaluation of medical devices — Part 1: Evaluation and testing within a risk management process

[3] I. Abad-Alvaro, D. Leite, D. Bartczak, S. Cuello-Nunez, B. Gomez, Y. Madrid, M. Armendia, M. Resano and. H. Goenaga-Infante, J. Anal. At. Spectrom., 2021, DOI: 10.1039/D1JA00068C.



Session 3: Advances in Measurement Applications

Wednesday 29th 10.00

Adventures in Elemental Mapping with Laser Ablation – ICP-MS

Simon Chenery¹, Charlotte Colvin², Eimear Deady³, Elliott Hamilton¹, Philip Hollyman⁴, Alicja Lacinska¹, Andrew Marriott¹

¹British Geological Survey, Nottingham, United Kingdom.

²Bangor University, Bangor, United Kingdom.

³British Geological Survey, Edinburgh, United Kingdom.

⁴British Antarctic Survey, Cambridge, United Kingdom

We are now several generations into laser ablation (LA) samplers coupled to ICP-MS's as elemental detectors. The latest of these generations, often making use of third-party software, will operate in an integrated fashion allowing easy, almost real time, elemental mapping. If appropriate standards are available, this mapping can be quantitative as well.

This presentation will describe how we went about optimising a new system (imageGEO193 LA from ESL + Agilent 8900 ICP-QMS and Iolite4 software). We considered competing requirements that clients request, such as smallest spots, most elements, best detection limits, maximum speed.

Optimised strategies were then tested in two application areas. The first aquatic biogeochemistry involved the analysis of shells and fish ear-bones, these grow daily with the chemistry of the hard part reflecting a complex mix of water chemistry, temperature and biophysical processes. The use of mapping showing real short-term variation that might be mistaken for noise using conventional single spot analyses.

The second area was more geological; the first application was investigating W/Sn/Sb/Zn mineralisation in SW England, mapping trace elements in various minerals, including wolframite. The aim being to determine if the mineralisation occurred in single or multiple phases and if the evolution of the original mineralising fluid could be elemental finger printed. A better understanding of mineralisation processes will allow identification of by-product (Ga, In in ZnS) or potential waste contaminants (As, Cd)



Session 3: Advances in Measurement Applications

Wednesday 29th 10.20

Exploring the use of nitrous oxide as a cell gas for inductively coupled plasma tandem mass spectrometry measurements

Shaun Lancaster¹, Thomas Prohaska^{1,2}, Johanna Irrgeher^{1,2}

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²University of Calgary, Calgary, Canada

Overcoming spectral interferences in inductively coupled plasma mass spectrometry (ICP-MS) measurements is of utmost importance when striving to achieve reliable data. One widely utilised method is the employment of a reaction cell gas to mass-shift either the analyte or the interfering species to obtain interference free determinations at the shifted mass of the analyte or on-mass. Amongst others, ammonia (NH₃) and oxygen (O₂) have been used as reaction gas to resolve interferences via the formation of molecular species in the reaction cell. However, NH₃ is a corrosive and toxic gas that is not universally compatible with every gas cell and O₂ only has a selective range of elements that react readily enough to benefit from mass-shift.

Nitrous oxide (N₂O) is an alternative reaction gas to form oxide species. N₂O has a much higher reactivity than O₂ and leads to increased sensitivity when used in ICP-MS. However, due to its high reactivity, a tendency to form new spectral interferences from other matrix components historically rendered N₂O unfavourable for ICP-MS measurements. With the advent of tandem mass spectrometry (MS/MS), formations of new interferences are no longer as problematic, as the additional quadrupole provides a mass-filter that only allows the target mass-to-charge ratio enter the reaction cell. Thus, N₂O becomes a more viable option and is recently gaining traction within the community.

Here, the use of N₂O has been explored as an alternative cell gas for ICP-MS/MS measurements. The comparison of reaction gases for selected elements of interest will be discussed.



Session 3: Advances in Measurement Applications

Wednesday 29th 10.40

Advances in stable and radioactive pollutant measurement using tandem ICP-MS/MS

Ben Russell¹, Emma Braysher¹, Hibaaq Mohamud¹, Heather Thompkins¹, Olivia Pearson¹, Frankie Falksohn¹, Ben Webster¹, Sharon Goddard¹

¹National Physical Laboratory, London, United Kingdom

The Nuclear Metrology (NM) and Air Quality and Aerosol Metrology (AQAM) Groups at the National Physical Laboratory (NPL) have combined plasma mass spectrometry expertise to advance capabilities for stable and radioactive isotope measurements using tandem inductively coupled plasma mass spectrometry (ICP-MS/MS). AQAM are responsible for measurement of stable isotopes in air filter samples as part of the Metals Network, covering 25 monitoring stations around the UK. NM have been using ICP-MS/MS to enhance measurement capabilities for radionuclides for decommissioning, environmental monitoring, nuclear forensics, nuclear medicine and underpinning nuclear decay data through updated half-life measurements.

This presentation will show how shared capability has led to projects where both groups have enhanced their capabilities, benefitting end users in multiple sectors. Examples will be shown of how ICP-MS/MS interference removal has led to measurement of radionuclides not previously measured e.g. Calcium-41, and where shared methods have been developed, such as for Nickel-63 measurement in radioactive waste samples, and stable Nickel isotopes for source attribution in metal network samples. Initial results will also be shown for testing of single particle mass spectrometry for Metals Network samples and nanoparticles from nuclear sites and for nuclear medicine applications. A new project on measurement of stable and radioactive pollutants as part of the European Green Deal will be introduced, for which NPL has a Work Package lead role. The project will develop harmonised methods and reference materials that will be of use for method testing and validation for all mass spectrometry instruments.



Session 4: Instrumental Advances

Wednesday 29th 11.30

Identification of ⁹⁰Sr in environmental samples via the hyphenation of ICP-MS with Collinear Resonance Ionisation Spectroscopy

Duggan M.¹, Flanagan K.¹, Edwards G.¹, Woodbury S.²

¹University of Manchester, Manchester, United Kingdom

²National Nuclear Laboratory, Central Laboratory, Sellafield, United Kingdom

The reported total volume of nuclear waste in the UK inventory is 4.56x10⁶ m³ with 94% of the volume classified as low-level waste, LLW, and very low-level waste, VLLW. However, the inventory of LLW-VLLW cross-boundary waste at 1.56x10⁵ m³ would benefit from an estimated saving of more than £1000 per m³ in disposal processes if the characterisation of radionuclides are correctly identified. The isotope ⁹⁰Sr, is chemically similar to calcium and upon consumption becomes a surrogate in bones leading to an increased risk of developing bone related cancers. Therefore, detection of ⁹⁰Sr abundance in the soil, groundwater and building materials near nuclear power plants serves to (i) reduce the cost of disposal due to correct identification of cross-boundary waste, and (ii) provide an accurate means of monitoring the abundance of carcinogenic radioisotopes in the environment. Both are of significant importance for the safe operation, maintenance, and decommissioning of a nuclear reactor site. The aim of the PhD project is to develop a rapid and cost-effective method for the detection of radionuclides from environmental samples. This is to be achieved by hyphenating the CRIS method with ICP-MS by designing and constructing a prototype ICP-MSCRIS unit that will provide proof-of-concept via the flagship isotope ⁹⁰Sr. Once the system has been validated it would then be optimised to identify various other radionuclides of interest to the nuclear industry.



Session 4: Instrumental Advances

Wednesday 29th 11.50

Work those quads – going the distance with Multi Quad ICP-MS

David Price¹

¹PerkinElmer, Seer Green, United Kingdom

The release of the NexION 5000 Multi Quad ICP-MS in 2020 further demonstrates how PerkinElmer continue to bring innovations to the field of atomic spectroscopy. This talk will introduce these technological improvements and provide examples of their successful use for the toughest of analytical challenges.



Session 4: Instrumental Advances

Wednesday 29th 12.10

10 years of ICP-MS/MS: How ICP-QQQ has changed ICP-MS applications and markets

Raimund Wahlen¹, Sonia North¹, Andrew Brotherhood¹

¹Agilent, Stockport, United Kingdom

Since the introduction of the world's first ICP-MS/MS system with the Agilent 8800 in 2012, MS/MS capabilities have opened up new analytical options across all markets where ICP-MS has been used for decades as well as significantly advancing the presence of ICP-MS for applications where it had not been used extensively previously.

The use of high-performance mass analysers capable of less than 1 amu resolution housed within high performance vacuum systems before the collision/ reaction cell allows a much wider range of analysis modes and cell gases (more than 15 have been reported to date) to be used for applications where abundant matrix components previously presented major challenges with interference removal and/ or method detection limits for both single-quad and sector-field ICP-MS instruments.

Since 2012 significant improvements in analytical capabilities have been widely reported using first- and second-generation Agilent ICP-MS/MS systems across all applications areas where ICP-MS is used with perhaps the most significant advances in areas such as geochemistry, nuclear, life-sciences, nanoparticle and materials analysis - including alloys and semiconductor - as well as more routine markets such as environmental, food and clinical.

The presentation will provide an overview of the most notable advances from a range of applications and illustrate the benefits of this technology that is now a routine analytical system in many laboratories rather than 'just' a highly capable technology for R+D purposes.



Session 4: Instrumental Advances

Wednesday 29th 12.30

How many Quads do you need? Choosing the right tool for right job.

David Bellis¹

¹SciMed, Stockport, United Kingdom.

ICP-MS/MS instrumentation have unquestionably increased the analytical capability and potential of ICP-MS in some specific applications. There is some confusion in nomenclature, IUPAC say 2 quads (ICP-MS/MS), some manufacturers say triple-quad and some say 4! (They are counting reaction cells and even an ion deflector). The PlasmaQuant MS from Analytik Jena has one quad, interference measurement is performed within the cone assembly. This allows re-focussing of the ion beam post-reaction/collision leading to some unique analytical benefits. Sensitivity is high and background very low giving amazing LODs in no gas, He collision, and H reaction modes. Mass shift using oxygen is also possible. The PlasmaQuant MS also operates with half the argon gas consumption of other instruments, including for high matrix samples. Whilst there are clear advantages of ICP-MS/MS for some application, the performance can often be matched or surpassed by a single-quad and applications should be selected on a fit-for-purpose basis.



Posters

Poster #1

Fast, high-resolution full elemental laser ablation imaging using time-of-flight ICP-MS for endogenous metal analysis and label identification in biological samples

Lukas Schlatt¹, Phil Shaw¹

¹Nu Instruments, Wrexham, United Kingdom

Bioimaging is a valuable tool to gain insight into the elemental composition of biological thin sections. Using the newest laser ablation techniques single bursts of data can be generated in a matter of a few milliseconds. This requires the fast detection to enable the fastest and most accurate possible recording of images. Since the analysis of as many elements as possible is typically required a TOF-ICP-MS becomes an important part of a modern LA-ICP-MS system for bioimaging.

The power of the Vitesse to record transient full mass spectra in sub-millisecond dwell times enables the full potential of the laser ablation systems creating large images at high resolutions in a matter of a few hours or even minutes. Furthermore, the full elemental detection enables the analysis of endogenous elements as well as higher mass isotopes often used as markers to further understand the metalomics and metabolomics in living organisms.

Data will be presented showing the latest images of biological materials highlighting the speed, resolution and sensitive detection of many elements. Spot sizes down to one micrometre were able to be used while still being able to ablate multiple square centimetres due to the modern ablation systems and fast acquisitions times. This allows an in-depth examination of endogenous elements for detailed analysis of the metabolome of biological samples on a cellular level.



Poster #2

Analysis of pyomelanin formation in vitro and in vivo to offer clinical insights

Hanaa Galeb^{1,2}, Adam Taylor¹, John Hardy¹

¹Lancaster University, Lancaster, United Kingdom.

²King Abdulaziz University, Rabigh, Saudi Arabia

Patients with alkaptonuria have elevated levels of homogentisic acid (HGA) and its derivatives in their bodies which leads to the deposition of pyomelanin nanoparticle pigmentation in specific bodily tissues. Here we report the analysis of polymerised HGA (polyHGA) nanoparticles formed under various conditions in vitro and in vivo. Various spectroscopic and microscopic techniques were employed to analyse pyomelanin nanoparticle formation in vitro, 1) in the absence/presence of different enzymes, 2) in the absence/presence of extracellular matrix components, 3) in the absence/presence of cells, and 4) in vivo using a turkey tendon model. A non-exhaustive list of analytical techniques employed include: UV-Vis, Fourier-transform Infrared (FTIR), Scanning Electron Microscopy & Energy Dispersive X-ray (SEM-EDX), Transmission Electron Microscopy (TEM), zeta potential measurements, Nuclear Magnetic Resonance (NMR), Electron Paramagnetic Resonance (EPR) spectroscopy, X-ray Photoelectron Spectroscopy (XPS); and the electrochemical properties assessed via Cyclic Voltammetry (CV) and Conductive Probe AFM (C-AFM) and scanning electrochemical microscopy (SECM). The study offers insight into the pigment formation process in vitro (e.g. effects of enzymes/ECM components) on pyomelanin formation and how that correlates to deposition in vivo, which offers molecular level understanding of potential clinical relevance.



Poster #3

Assessment of occupational exposure to hexavalent chromium – recommendations from HBM4EU chromate study Introduction

Elizabeth Leese¹

¹HSE Science and Research Centre, Buxton, United Kingdom

Hexavalent chromium (Cr(VI)) is an important occupational carcinogen. In addition to air monitoring, biomonitoring is commonly applied to monitor exposure to Cr(VI). Within the EU biomonitoring initiative, HBM4EU, we explored the applicability of different biomonitoring methods in the assessment of occupational exposure to Cr(VI) in welding and surface treatment activities.

A multi-center cross-sectional study was performed in Belgium, Finland, France, Italy, Poland, Portugal, the Netherlands, Luxembourg and United Kingdom. Harmonized procedures were used to collect biological and industrial hygiene samples. Contextual information was collected using questionnaires. Altogether 602 exposed workers and controls were included in the study. Exposure biomarkers studied included urinary, red blood cell and plasma chromium, and exhaled breath condensate speciation of Cr(VI)/Cr(III). In addition, a number of effect biomarkers were studied.

All exposure markers showed highest exposure levels among chrome plating workers. Urinary chromium showed a good correlation with air Cr(VI) in both platers and welders. Observed low correlations between different exposure biomarkers suggest that these approaches are not interchangeable but rather complementary.

In conclusion urinary chromium showed its value as the first approach for the assessment of internal exposure to Cr(VI). At low exposure levels paired samples are recommended. Chromium in red blood cells and plasma as well as chromium species in exhaled breath condensate were shown to provide additional information when more specific information on exposure is needed.



Poster #4

Accurate identification, examination and differentiation of multielement nanoparticles using time of flight ICP-MS and sub-millisecond spectral acquisition times

Lukas Schlatt¹, Phil Shaw¹

Nu Instruments, Wrexham, United Kingdom

The use of nanoparticulate materials has been increasing with large amounts being released into the environment each year. Therefore, sensitive methods are needed to detect these particles. While the size detection limit has been decreasing over the years with more sensitive detection methods becoming available, completely new instrumentation to scanning mass spectrometers is necessary to examine the composition of individual particles.

The Vitesse TOF-ICP-MS can measure the full mass spectrum continuously at acquisition speeds down to 80 μ s. Together with powerful peak integration and identification methodologies, the data from individual nanoparticles can be identified and reduced efficiently and easily. In addition to the capability to record multiple full mass spectra for every particle, examinations of nanoparticles with a reduced mass range can be recorded to gain a higher sensitivity for a smaller number of observed isotopes. This allows for the reduction of size detection limits and more detailed examination of elemental ratios in single nanoparticles.

Data is shown, which differentiates a mix of various particles into clusters. This way a mixture containing various similar as well as different particles can be differentiated quickly and easily. Furthermore, the advantage of reduced mass range examinations is shown and discussed.



Poster #5

A Not so Rare Element: Gadolinium as an Interference on Selenium in Clinical Samples.

Chris Harrington¹

¹SAS Trace Element Laboratory, Guildford, United Kingdom

Measurement of selenium in serum is an important clinical biomarker of nutritional status. The presence of gadolinium (Gd) in samples following administration of the contrast agents used for Magnetic Resonance Imaging, results in a significant positive bias in using ICP-MS.

We report on the methods available to eliminate this interference and the results of an inter-laboratory external quality assurance exercise, to assess the current best practice in clinical measurements.



Poster #6a

Stanley Greenfield's contributions to analytical chemistry: 1. atomic spectrometry

Michael Foulkes¹, Steve Hill¹, James Miller², Helen Reid², Maryanne Thomsen³, Alan Townshend⁴, Julian Tyson⁵

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²Loughborough University, Loughborough, United Kingdom.

³Agilent, Leicester, United Kingdom.

⁴University of Hull, Hull, United Kingdom.

⁵University of Massachusetts, Amherst, USA

Professor Stanley Greenfield (1920 – 2019) was maybe the first scientist in the world to recognize the potential of the inductively coupled plasma (ICP) as an atom reservoir for spectrochemical analysis. In the early nineteen sixties, his research group at Albright and Wilson (in Oldbury, England) built an atomic emission spectrometer with an ICP source [1] that, compared with AAS, was free from stable compound formation, had superior sensitivity, and detected a wider range of elements. They reported that the source could accept the direct introduction of powders and slurries as well as liquids, and was, of course, free from contamination by electrode materials. Perhaps, most importantly of all, they recognized that their plasma was annular, with a cooler central channel through which the sample passed. Encouraged by the analytical faculty at Birmingham University (where he was a visiting lecturer), Stan then vigorously advocated for the ICP as a source for spectrochemical analysis through numerous publications and conference presentations. Following retirement in 1980, he set up shop as Industrial Professor at Loughborough University, where he pursued his idea of constructing an AFS instrument with two ICPs, one as the atomizer and the other as the light source [2]

1. S. Greenfield, I. Ll Jones and C. T. Berry, High-pressure plasmas as spectroscopic emission sources, *Analyst*, 1964, **89**, 713 – 720.
2. S. Greenfield and M. Thomsen, Atomiser, source, inductively coupled plasmas, in atomic fluorescence spectrometry (ASIA). Some recent work, *Spectrochim. Acta Part B*, 1985, **40**, 1369-1377.



Poster #6b

Stanley Greenfield's contributions to analytical chemistry: 2. atomic spectrometry (continued) and the AD/RSC

Julian Tyson¹

¹University of Massachusetts, Amherst, USA

Following retirement from Albright and Wilson in 1980, he set up shop as Industrial Professor at Loughborough University, where he pursued his idea of constructing an AFS instrument with two ICPs, one as the atomizer and the other as the light source [2] and his interests in microwave-assisted sample preparation [3]. He continued his interests in the possibilities of novel modes of excitation in ICP-AFS [4] in a subsequent collaboration at the University of Plymouth [5]. Further details of his extensive contributions to both analytical atomic spectrometry (with a brief mention of his work on gas analysis, the condensed phosphates and chemiluminescence) and to his wide-ranging involvement with the Analytical Division of the Royal Society of Chemistry [6], notably though the work of the Analytical Methods Committee, will be presented.

1. H. J. Reid, S. Greenfield and T. E. Edmonds, Investigation of decomposition products of microwave digestion of food samples, *Analyst*, 1995, **120**, 1543 – 1548.
2. S. Greenfield, T. M. Durrani, J. F. Tyson and C. A. Watson, A comparison of boosted-discharge hollow-cathode lamps and an inductively coupled plasma (ICP) as excitation sources in ICP atomic fluorescence spectrometry, *Spectrochim. Acta Part B*, 1990, **45**, 341-49.
3. A. Young, L. Pitts, S. Greenfield and M. Foulkes, A preliminary comparison of radial and axial excitation fluorescence in the ICP using non-laser sources, *J. Anal. At. Spectrom.*, 2003, **18**, 44-48



Poster #7

The effects of feeding seaweed to dairy cattle on methane production and milk iodine content

Ali Alzahrani¹, Asta H Petursdottir², D Humphries³, S Lignou⁴, C Fagan³, M rodehutscord⁵, C Reynolds³, H Gunnlaugsdóttir², Rainer Ebel¹, Gareth Norton⁶, Joerg Feldmann⁷

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³School of Agriculture, Policy and Development, University of Reading, UK.

⁴Department of Food and Nutritional Sciences, University of Reading, UK.

⁵Institute of Animal Science, University of Hohenheim, Stuttgart, Germany.

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Seaweed has gained attention for its ability of some species to reduce methane emitted from cattle. Seaweed is rich in iodine and has been shown to increase the iodine content in milk from dairy cattle fed brown macroalgae.

Based on previous in-vitro evaluation of methane inhibition by a variety of seaweeds in the SeaCH4NGE project (www.eitfood.eu), the objective was to measure the effects of feeding mixtures of seaweed on the methane production from dairy cattle and the iodine concentration of their milk and milk products. 40 lactating Holstein dairy cows were fed one of four treatment diets using *Ascophyllum nodosum* (AN), *Fucus vesiculosus* (FV) and *Asparagopsis taxiformis* (AT). Seaweed inclusion rates in the diet were as follows: A (0.15% AN:FV:AT), B (1.75% FV:AN), C (control, no seaweed added) and D (0.65% AN:FV). The limiting factor of the inclusion rates was the iodine concentration of the seaweeds, where groups A, B and D received iodine close to the maximum permitted level of iodine in cattle diets. Treatments A, B and D increased milk iodine concentration ((170%, 180% and 216% higher I compared to control, respectively). Iodine in milk products such as butter, UHT milk and pasteurized milk also showed an increase in iodine compared to control when seaweed was fed (166%, 243% and 155% respectively). However, treatments B and D had no effect on methane production. For treatment A the reduction observed was 9% of methane production and 12% of methane per kg feed dry matter intake.



Poster #8

Resolving the unresolved: online microdialysis coupled to ICP-QQQ for the simultaneous sampling and analysis of dissolved elements in soil solution

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Assessing rapid chemical-elemental reactions in soils is significantly inhibited by the spatial and temporal resolution of current sampling techniques [Rhizon™ samplers, diffusive gradients in thin films (DGTs)]. Soil chemistry is typically investigated over hours-days-weeks and with poor sampling density; the vast majority of reactions occur within seconds-minutes.

Microdialysis (MD) is a new technique in the field of soil science that uses small probes to sample compounds dissolved in soil solution, with minimal disturbance to external environments. Initially developed for use in neuroscience, MD has the potential for translation to environmental geochemistry to define soil chemical/physical parameters, and better inform predictive models for soil-to-plant transfer of potentially harmful elements (PHEs) or essential nutrients. One considerable experimental challenge for MD is balancing the target analyte recovery efficiency with the sample volume required for the analytical chemistry technique, which can significantly affect how often elemental speciation changes and soil fixation events can be measured.

To overcome this challenge, we have begun development of a novel integrated online MD sampling and analysis technique, through direct coupling of MD probes with triple quadrupole inductively coupled plasma mass spectrometry (ICP-QQQ) using a microflow total consumption nebulizer with no additional modifications. This poster will present the initial setup, optimisation and application of the technique to the sampling and analysis of multiple elements in soil solution, alongside future perspectives on how information gained from this promising technique can contribute to the management of global societal and agricultural issues (e.g. nutrient supply to staple crops, contaminated land remediation).



Poster #9

Observation of high concentrations of unidentified organofluorine in pilot whales stranded in Scotland

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Per- and polyfluoroalkyl substances (PFASs) are a group of anthropogenic chemicals, produced since the 1950s. These chemicals when applied on the surfaces of various products make them resistant to moisture, stain, or stickiness. Global detection of these compounds in the environment, and human, and wildlife matrices has increased. The increasing use of such compounds has sparked a lot of environmental concerns since they degrade quite slowly. Exposure to PFAS has been linked to a variety of adverse health effects in humans and wildlife, including immunotoxicity, developmental problems, and cancer. Although new methods are being developed, analyses of PFASs in biota at low concentration levels pose a significant analytical challenge. In this work combustion ion chromatography for total fluorine was applied to quantify extractable organic fluorine (EOF) in liver tissue of pilot whales stranded in Scotland and high-performance liquid chromatography-tandem mass spectrometry (HPLC-MS/MS) was used for known PFASs in a targeted analysis. Comparisons between the amounts of known PFASs and EOF in the livers showed that a large proportion of the organic fluorine in the liver is of unknown origin. Similar PFAS profiles were observed in liver tissue over the different ages and the proportion of unknown compounds increase with age.



Poster #10

ICP-OES Options for Improving Data Quality for Complex Samples

Andrew Brotherhood¹, Rai Wahlen¹, Sonia North¹

¹Agilent, Stockport, United Kingdom

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Poster #11

Options for Improving Sample Throughput & Reducing Operating Costs

Andrew Brotherhood¹, Rai Wahlen¹, Sonia North¹

Agilent, Stockport, United Kingdom

With rising gas and energy costs, it's more important than ever to control your laboratory's expenditure. Find out how Agilent's reliable and easy to maintain sample introduction valve systems can help you reduce costs and analyse many more samples per hour.



Poster #12

The preservation of total dissolved mercury from water samples using functionalised C18 solid-phase extraction

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Due to its persistent and bioaccumulative properties, mercury is considered one of the most toxic elements to human health, and its species are found in all spheres of the environment. Since the widespread poisoning event in 1968 in Minamata, Japan, the monitoring of mercury pollution is of utmost importance, particularly where the risk of human exposure is high.

The preservation of mercury species in water samples remains a challenge. The recommended preservation procedures are unsuitable for many locations, suggesting shipping of water samples overnight to laboratories, collection of samples greater than 500 mL or acidification with hydrochloric acid and analysis within 48 hours. This may not always be possible, particularly in remote areas or in developing countries where analytical sensitivity may not be suitable for mercury analysis. Solid-phase extraction offers a method to extract mercury from water samples and preserve speciation data. These techniques are already employed in mercury analyses, often for the preconcentration of the metal and for online speciation. Solid-phase extraction offers a potential cost-effective method of preserving mercury speciation data that can be used for many aquatic matrices.

A method for preserving total mercury was developed using dithizone functionalised C18 cartridges, allowing for transfer from field to laboratory while maintaining representative dissolved mercury concentrations. The method incorporates a preconcentration of the dissolved mercury, improving detection limits, and has little to no associated risks to the operator in the field.



Poster #13

The use of time of flight ICP-MS and very fast washout laser ablation systems to accurately image major and minor isotopes as well as elemental ratios in geological samples

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The ability for multi-elemental analysis in solid samples using rapid and direct methods is a key point in the development of analytical science. Over the years, in situ techniques based on Laser Ablation and Inductive Coupled Plasma Mass Spectrometry (LA-ICP-MS) have been widely used rapidly evolving into well-established, mature powerful tools for direct, high sensitivity and precision, and high lateral resolution analysis in numerous fields such as geology, biology, metallurgy, environmental sciences, etc. Significant research and advances continue to thrive to achieve the fastest, most accurate and efficient analysis.

The fast acquisition speeds of the full elemental mass spectrum make a TOF-ICP-MS the most appropriate instrument for the fast examination of high-resolution elemental maps using the newest laser ablation systems. The often mentioned downside of a TOF-ICP-MS is its limited dynamic range, especially if all examined isotopes have to be examined. The Vitesse allows for beam attenuation, enabling a novel technique to create images showing elements between 100% and sub-ppm levels in one image. Examples of this technique will be shown and normalization techniques discussed.

Furthermore, the Vitesse can be tuned for the optimal detection of isotope ratios. Examples showing the differentiation of lead uranium ratios in Zicons at low micrometer resolutions will be shown. Combining the various acquisition methods in an automated fashion allows for the detailed examination of various samples in a short time with minimal input from the user. Finalized data can be shown live, or exported to import into other software packages.



Poster #14

Determination of metals generated during the thermal failure of lithium ion batteries

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Lithium ion cells, although near ubiquitous as a portable power source in today's society, have rare, but well documented failure pathways which generate gas, fume and smoke, and often result in fire. Whilst the composition of the gas has been subject to much analysis, far fewer reports have focussed on the nature of any solid materials released.

This work describes the causing to fail (by applying an external heat source) of a range of commercially available and widely used cells. Samples of both the smoke generated during the failure, and the residues left surrounding the cell after failure, were analysed for metal content by ICP-AES. These showed that all samples contained the key metals expected to be in cell cathodes (nickel, manganese, cobalt and aluminium) in not dissimilar ratios. However, the ratio of these elements differed from cell to cell, as the cathode varied with results suggesting that that the cathode metals dominate/mimic those seen in the smoke.

These findings suggest that it may be possible to identify the battery devices that have been involved in a fire event.

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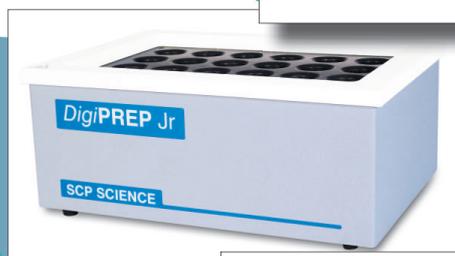
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