

BNASS 2018, St Mary's University, Strawberry Hill, 2nd -4th July 2018

Instructions For Poster Presentations

- Posters will be displayed in the Students Union Hall (building J on campus map).
- The poster boards are 2m high by 1m wide and A0 sized posters in portrait orientation will fit comfortably.
- Velcro fasteners will be provided; no pins or staples should be used.
- Posters should be mounted by 13:00 on Monday July 2nd.
- Posters for **Monday's session** (16:30-17:30) should be placed on **even numbered** boards and those for **Tuesday's session** (15:05-16:00) on **odd numbered** boards.

Monday Poster Session 1: Biomedical, Quality Assurance and Instrumentation

Exact determination of Ferritin as a potential marker for Alzheimer's disease by ID-ICP-MS

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According to estimates over 6 million people are affected in the European Union by neurodegenerative diseases which pose a big challenge to the health care systems. In an ageing population the diagnosis and treatment of these diseases are gaining more and more importance. With about 70% the most common form of dementia is Alzheimer's disease (AD). Determination of the biomarkers for AD (most established are β -amyloid peptide and τ -protein) is generally performed using immunoassays or optical methods which often lead to incomparable results. Besides the established biomarkers, metalloproteins are under discussion as potential clinical markers. For example, ferritin (FER) levels in cerebrospinal fluid (CSF) are in correlation with brain iron levels which are increased in AD. A promising approach for the quantitative determination of FER via its S-content in CSF is species-specific isotope dilution inductively coupled plasma mass spectrometry (SS-ID-ICP-MS) as it is a primary measurement method and gives results that are traceable to the International System of Units (SI). To perform SS-IDMS, a protein reference and an adequate spike material are needed. As there is no FER reference material commercially available, pure native FER is characterized in-house for the use as reference. A recombinantly produced FER enriched in ^{34}S acts as spike.

Acknowledgement: This project has received funding from the EMPIR programme co-financed by the Participating-States and from the European Union's Horizon 2020 research and innovation programme.

Simultaneous quantification of Fe and Se, at clinically relevant concentrations, in brain tissue using LA-ICP-MS: tissue homogenate preparation and Se signal enhancement

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Disruption to iron homeostasis at regional or cellular levels in the brain has been implicated in excessive formation of reactive oxygen species (ROS)¹. This can lead to abnormal A β peptide aggregation and the formation of senile plaques resulting in cell death, which are hallmarks of neurodegenerative diseases.

Contrastingly, selenium containing proteins have antioxidant properties and play a role in neuroprotection and regulation of these ROS².

Due to the contrasting roles played by Fe and Se in the brain there is an increasing interest in the ability to quantitatively map these elements simultaneously in micro-sections of brain tissue. However their natural concentrations in the brain vary from low ppb for Se to hundreds of ppm for Fe. This causes many analytical challenges; such as the lack of sufficiently stable and homogeneous matrix-matched calibration standards and the large concentration gap between co-located Fe and Se.

This work describes an optimised protocol for producing tissue homogenates spiked with inorganic Fe and Se and/or their high molecular weight species including Ferritin and Selenoprotein P. Main considerations to produce calibration standards, which are sufficiently both stable so can be re-used from batch to batch and, homogeneous (RSD of 15% and 19% for Fe and Se, respectively) will be highlighted. Finally, method development to increase the signal-to-noise ratio for Se using ICP-carbon addition approaches will be discussed.

1 J. F. Collingwood and M. R. Davidson, *Front. Pharmacol.*, 2014, **5**, 191.

2 D. Xiubo *et al.*, *Curr. Top. Med. Chem.*, 2016, **16**, 835

Chronic kidney disease of unknown origin in sugarcane industry: metals analysis in biological samples

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In Central America, there is an epidemic of Chronic Kidney Disease of unknown cause (CKDu), also referred as Mesoamerican Nephropathy (MeN). In the last 15 years it has been estimated that CKDu has caused the premature death of approximately 20,000 individuals, mainly young males, who work in physical intensive manual labour, hot climate agriculture such as sugarcane cultivation and sugar production. Sufferers reach end stage renal disease before the age of fifty. Over the last two decades there has been a progressive increase in CKDu and the work presented here is part of a study investigating different possible causative factors.

Following site visits to different locations in Nicaragua, urine samples were collected from 330 workers at both the start and end of the harvest season. This include of workers who did and did not exhibit kidney function decline, as well as from two different geographical locations.

In this part of the project a range of elements were analysed in the urine samples by ICP-MS. Whilst the results from the metals analysis showed elevated urinary aluminium, cadmium, copper and silicon levels, no correlation with kidney function status was observed.

As yet there is still no clear evidence as to what is the leading cause of kidney failure and multiple pathways have been proposed. The levels of elements determined here provide an association with metals exposures however this is not in every worker and more work is needed to understand this further.

Total element determination in the brains of different mouse models of Alzheimer's disease, by ICP-MS.

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Alzheimer's disease (AD) is the most common cause of dementia, and is a growing health concern worldwide, with no cure or disease modifying therapy. It is pathologically characterised by the presence of neurofibrillary tangles of tau protein, and β -Amyloid plaques within the brain, however, evidence also suggests that metals play a role in disease mechanisms. This work used ICP-MS to determine the total element concentrations of various elements within the brains of four different mouse models: 3 AD disease models and one Wild Type (WT) control. The disease models overexpressed Tau and β -Amyloid, with varying levels of gene expression and pathological development. This work used pig brain for method development and aimed to establish if pig brain would have potential as a reference material for an inter-laboratory study, with a reproducibility of 10%). This talk will present data gathered, and discuss possible reasons for changes in elemental concentrations.

Multi-element determination in populations of single cells by quadrupole ICP-MS

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Recent advances in analytical technology enabling the analysis of single cells has resulted in increased interest in this field of research. Previously, elemental quantification in life science research has been performed by a series of processes including culturing large numbers of cells, preparing cell extracts, and defining analyte concentrations as an average value in cell extractions. However, this method assumes that the cell population is homogenous, and therefore might miss minority cell clusters which exhibit significant differences. In contrast, single cell analysis allows cells to be injected individually, enabling us to understand the detailed elemental profiles of cell clusters. Single cell analysis using ICP-MS is performed by suspending cells in solution to separate them, vaporizing and ionizing the cells using an Ar plasma and detecting the resulting ion cluster as a signal pulse at the ICP-MS detector lasting less than 1 msec. Elemental concentrations in the cells are determined from the peak areas. We have developed a new sample introduction system comprising a total consumption nebulizer and spray chamber which greatly improves the transmission efficiency of μm size particles and has enabled us to quantify the concentrations of P, Ca, Fe, and Zn at ag/cell level in bakery yeast cells (*Saccharomyces cerevisiae*). Newly developed software monitors 16 elements within the same batch, and calculates the analyte concentrations in the cells automatically. In this presentation, we will show the fundamental results based on the new hardware and software development for the single cell analysis.

EURACHEM: A Focus for Analytical Chemistry in Europe

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Eurachem (www.eurachem.org) is a network of organisations within Europe designed to a) establish a system for the international traceability of chemical measurements and b) promote good quality practices in analytical sciences. Currently represented in 33 European countries, it aims to provide a forum for analytical scientists, laboratory staff and those interested in using the results of analytical measurements, to discuss common problems and develop informed and considered approaches to both technical and policy issues.

Members and stakeholders meet once a year at the Eurachem General Assembly. An Executive Committee and several topical Working Groups pursue the organisation's stated goals throughout the year, often in cooperation with other organisations. Participation is open and channeled through national representatives.

Eurachem's main output is authoritative guidance documents, promoted through dedicated events which are also designed to provide opportunities for collecting feedback. Beside the guides, information leaflets, *i.e.* short briefing documents on specific topics, are intended to inform a wide audience, including laboratory staff, managers and laboratory customers. This poster, presented by the members of the Eurachem Executive Committee, aims to summarise current Eurachem activities, inform readers about the available guidance and attract active participation.

Reliable Estimates of Measurement Uncertainty Obtained from External Quality Assessment Schemes: A Case Study

Marina Patriarca¹, Valeria Patriarca¹, Josiane Arnaud², Monserrat González Estechea³, Chris Harrington⁴, Samantha Shepherd⁵, Montserrat Ventura⁶, M^aCarmen González⁶, Cas Weykamp⁷

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The estimate of measurement uncertainty (MU) is a requirement for the competence of testing, calibrating and clinical laboratories (ISO/IEC 17025, ISO 15189). The Nordtest approach provides a model for robust MU estimate applicable in single, routine, analytical laboratories using information collected from the procedures in place for the quality assurance of results. In laboratory medicine, external quality assessment schemes (EQAS) typically consist of numerous samples to be analysed at regular intervals over a given period and including duplicates unknown to the participants. Beside scoring performance on the individual items, these data can be exploited to provide the information needed for the estimate of MU according to the Nordtest approach.

In this study, data from EQAS for lead in blood, designed to provide participants with 24 items including 12 unknown duplicates, over a cycle of one year, were analysed to determine laboratory intermediate precision and bias variability. These components were combined to determine the MU of the laboratory results over the concentrations range. The reliability of this approach was successfully tested using the individual estimates of MU to calculate zeta-scores (a type of score where the deviation from the assigned value is compared with the MU of the laboratory result) for the results provided by the same participants in the first six months of the following EQAS cycle. This approach provides robust estimates

of MU from data already available as part of EQAS and allows laboratories to fulfil the need for periodic review of such estimates.

The use of External Quality Assurance in improving the methods used for clinical trace element analysis in liver biopsy samples

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Running since 2012-13 the solid matrix EQA scheme provides specimens for laboratories that undertake the analysis of solid samples for copper and iron. The scheme is offered by UK NEQAS for Trace Elements in Guildford to 18 participants worldwide.

Clinically, in patients suspected of having Wilson Disease or genetic haemochromatosis, a liver biopsy sample (<5 mg) may be taken for the determination of copper or iron respectively, to aid diagnosis. To ensure accurate testing methods in these complex determinations this scheme sends out small mass (10 - 20 mg) powdered samples of animal organs or other suitable organic solid materials, which are analysed by the participants for their copper and iron content. The analytical procedure used requires the organic materials sent to be converted to a liquid prior to analysis; this step can be carried out in a number of ways using various combinations of reagents and heating methods. It was felt that this complex step lead to significant errors being introduced due to contamination or under-recovery of the analyte from the matrix.

Over the last year and a half, in addition to the solid samples analysed, the laboratories also tested pre-digested samples prepared by the EQA program. Some samples were spiked to concentrations which better reflect clinically relevant levels (Reference ranges in tissue: Cu >250 ug/g, Fe > 1508 ug/g dry weight). This study aims to determine the source of the error and attempt to offer advice on best practice.

Development of new certified reference materials for SI-traceable $^{13}\text{C}/^{12}\text{C}$ isotope amount ratio measurements

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Reference materials (RMs) play a key role in calibration of isotope ratio mass spectrometers, assigning property values and the transfer of measurement accuracy between different laboratories and over time. A long-standing problem associated with the latter function of isotopic RMs stems from the fact that many RMs used in differential, or delta value, isotope ratio measurements lack a straightforward link to the International System of Units (SI). Instead, traceability in delta value isotope ratio measurements is established to scale-defining artefacts which are recognised as International Standards and represent the end members of the traceability chain for the measurements.

$\delta^{13}\text{C}$ delta measurements are a good example of a situation where it gets increasingly more difficult for practitioners to achieve comparability of measurement results. Establishing traceability of $\delta^{13}\text{C}$ delta measurements to the SI as a stable metrological reference system can be a sustainable solution for ensuring comparability of measurement data. In this work, we have produced and characterised three new glycine RMs certified for $^{13}\text{C}/^{12}\text{C}$ isotope amount ratios and $\delta^{13}\text{C}_{\text{VPDB}}$ delta values, covering a wide range of $\sim 54\text{‰}$ ($^{13}\text{C}/^{12}\text{C}$ isotope amount ratios with expanded uncertainties in parentheses are

0.010642(30), 0.010821(30) and 0.011227(32), respectively). $^{13}\text{C}/^{12}\text{C}$ isotope ratio measurements were performed by both multicollector inductively coupled plasma mass spectrometry (MC-ICPMS) and flow injection analysis-chemical oxidation-isotope ratio mass spectrometry (FIA-CO-IRMS) using a calibration strategy based on the use of synthetic isotope mixtures gravimetrically prepared from well characterised, highly ^{13}C -enriched and ^{13}C -depleted glycines. New RMs will be invaluable in improving comparability of $\delta^{13}\text{C}$ measurements.

Determination of total fluorine of organofluorine compounds in cookware using high-resolution graphite furnace molecular absorption spectrometry.

Abdullah Akhdhar, Tengetile Nxumalo, Laili Jamari, Eva Krupp, Joerg Feldmann

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Fluorine has a very high ionisation potential of 17.42 eV that has a high tendency to obtain a negative charge (1). Fluoride is an essential for human health and can protect from dental decay and promotes healthy bones. Humans are exposed to fluoride through food and drinking water and by breathing air (2). According to World Health Organization, the recommended limit of fluoride in drinking water between 0.8 and 1.0 mg/L (3). European Food Safety Authority has recommended the fluorine intake 0.05 mg/kg of body weight per day for children and adults (4). Perfluorinated compounds (PFCs) are xenobiotics and have been detected in the blood of wildlife and humans worldwide. They pose great threats to human health due to its persistence and bioaccumulative effects, PFCs have become the hot topic in recent years (5, 6). PFCs have found in cookware, fast-food containers, carpet cleaning solutions, beverage cups, and microwave popcorn bags (7). In this study, the absorption of calcium mono-fluoride (CaF) molecule has been applied for the determination of fluorine of organofluorine compounds and high-resolution continuum source graphite furnace molecular absorption spectrometry has been employed. The rational line at 606.432 nm was used for measuring the molecular absorption (1). The pyrolysis and atomization temperatures were 500 °C and 2100 °C. The method was used to determine the total fluorine rather than fluoride, which includes polar organofluorine and nanoparticles. The values were found in cookware samples by using water extraction between 534 and 1900 ug/L

Improved data acquisition speeds for high resolution ICP-MS

Phil Shaw

Nu Instruments, Wrexham, United Kingdom

High resolution magnetic sector ICP-MS has become the favoured instrument for many laser ablation applications due to the high sensitivities seen in dry aerosol sampling and the flat topped peak characteristic of the magnetic sector mass spectrometer.

Recent improvements to laser ablation sample cells have prompted requests for even faster data acquisition for wide if not full mass spectra, without sacrificing the sensitivity benefits of the magnetic sector when used in dry aerosol mode. This work will describe new data acquisition modes available with the recently released fast magnet power supply and controller. It has been possible to increase the magnetic field ramping and fall-back speeds to obtain spectra up to uranium in 105ms leading to an acquisition rate of 9.5Hz.

With a fixed magnet cycle time, the acquisition time of each mass is governed by the number of isotopes chosen for the method and the allowable range of the deflectors used to jump from one isotope to another. This work will describe the “LinkScan” technique which has been used for many years

successfully in multiple laser ablation labs and the modification of the deflector scan range made to improve the duty cycle further for this technique.

Laser-Induced Breakdown Spectroscopy of Actinides: Elemental and Isotopic Analysis for Nuclear Forensics

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Laser-induced breakdown spectroscopy (LIBS) permits real time elemental analysis of a material irrespective of its nature, physical state or environment. The ability to perform contact-free or stand-off analysis of materials by this method offers significant advantages for applications which require the interrogation of hazardous materials contained within controlled or inaccessible environments. This capability is a particularly attractive feature for applications in the nuclear industry and in the field of nuclear forensics. The LIBS technique demonstrates many of the attributes required of a field-deployable method: these include accurate and rapid analysis, no (or minimal) sample preparation and the ability for remote operation. Whilst the technique is capable of identifying all elements in a sample on the basis of their atomic / ionic emission spectra, applications in isotopic analysis present a greater challenge and are only feasible for elements at the extremities of the periodic table (e.g. hydrogen and uranium). Furthermore, isotopic LIBS requires the use of high resolution spectrometers which have poor light throughput and lack portability.

This presentation will focus on the development of a system based on the use of tuneable diode laser absorption spectroscopy (TDLAS), in conjunction with LIBS, for isotopic analysis of uranium. This 'hyphenated' approach (LIBS-TDLAS) will provide information regarding the elements present in the samples of interest by LIBS whilst TDLAS will be used to ascertain the $U^{235}:U^{238}$ isotopic ratios. This approach offers potential applications in nuclear forensics investigation as it can provide information on the provenance of materials containing uranium (e.g. uranium ore concentrates and processed material). The feasibility of the LIBS-TDLAS approach has been demonstrated by detecting lithium in a laser-produced plasma using diode laser absorption spectroscopy at 670.78 nm.

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Improving the accuracy of nuclear decay data using tandem inductively coupled plasma mass spectrometry (ICP-MS/MS)

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The accuracy and precision of nuclear decay data underpins all applications of nuclear science, including radiometric dating, climate modelling, nuclear medicine and decommissioning of nuclear sites. However, many of these data were collected decades ago and large uncertainties remain, which limits the measurement precision achievable. Advances in mass spectrometric methods can be combined with established decay counting techniques to reduce the uncertainties in nuclear decay data of multiple long-lived radionuclides, offering benefits to a range of end users.

This project presents the methodology for measurement of half-life values for long-lived radionuclides using a combination of decay counting and ICP-MS/MS. This includes determination of isotopic composition, as well as weighing of the source material and measurement of the activity concentration. Isotope dilution techniques allow for the measurement of the relative abundance of radionuclides by

spiking of a reference solution, eliminating errors related to signal intensity and is therefore a useful technique in such high-precision isotope analysis.

The aim of this project is to improve the accuracy of half-life values for priority long-lived radionuclides, focusing on naturally occurring ^{238}U and ^{232}Th . Both these radionuclides have geochronological applications involving large timescales, in which changes to the uncertainty of decay data are significant. The methodology developed will be applicable to other natural and anthropogenic radionuclides that suffer from similar decay data limitations.

Tuesday Poster Session 2: Elemental Speciation, Nanoparticulates, Environmental and Food Analysis

A METROLOGY STRATEGY FOR THE ACCURATE DETERMINATION OF CHROMIUM (VI) IN WATER USING HPLC-ICP-MS WITH SPECIES-SPECIFIC ISOTOPE DILUTION ANALYSIS.

John Entwisle, Sarah Hill, Panayot Petrov, Heidi Goenaga-Infante

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Trivalent [Cr(III)] and hexavalent [Cr(VI)] chromium are commonly observed species in the environment and their redox state is known to interconvert. Cr(VI) is highly toxic and recognised as a carcinogen. Therefore, procedures for the SI traceable quantification of Cr(VI) in the presence of the other Cr species are required.

In this work, a reversed phase HPLC separation method that enabled baseline separation of Cr(III) and Cr(VI) within 15 minutes was developed with Cr isotope detection by ICP-MS. Quantification of Cr(VI) was undertaken by species-specific double spiking single isotope dilution with HPLC-ICP-MS. To achieve this, $^{50}\text{Cr(III)}$ and $^{53}\text{Cr(VI)}$ -enriched spikes were produced and characterised *in-house*. Using this method, the Cr(VI) mass fraction of a water sample from the Aquacheck Proficiency Testing (PT) Scheme was found to agree well with the assigned value.

The same separation method was used in combination with species-specific double isotope dilution analysis and this measurement capability was demonstrated through participation in an international inter-laboratory key comparison study (CCQM K124) for CrVI in drinking water. Spiking, dilution (1:6) and stabilisation were achieved in one step by the gravimetric addition of an appropriate amount of $^{53}\text{Cr(VI)}$ to the sample to achieve a target isotope ratio of 1 for $^{52}\text{Cr(VI)}/^{53}\text{Cr(VI)}$. A relative expanded uncertainty of 1.42% ($k=2$) was achieved for Cr(VI) at $62.88 \pm 0.90 \mu\text{g kg}^{-1}$. The value reported by LGC was in good agreement with the key comparison reference value.

Quantification and Identification of Perfluoroalkyl Compounds using HPLC simultaneously coupled to ICPMS/MS and ESI-Orbitrap-MS

Tengetile Nxumalo, N. Laili A Jamari, Abdullah Akhdhar, Andrea Raab, Eva M. Krupp, Jorg Feldmann

University of Aberdeen, Aberdeen, United Kingdom

Perfluoroalkyl compounds (PFCs) have been detected in many different environmental samples. A trend which has been noted is that the commonly measured PFC species in different environmental samples account for only a small fraction of the extractable organofluorines present in these samples. These unknown fluorinated organic compounds, which might be abundant in the environment present a pronounced basis of uncertainty for ecological and humans health risk assessment, and thus their identification and characterisation is vital. The main challenge in the identification of these fluorinated compounds is the lack of a fluorine specific detector. In this work we show the use of ICP-MS/MS as a fluorine specific detector for chromatography to determine PFCs individually. PFC species were separated using reverse phase high performance liquid chromatography (RP-HPLC) and simultaneously detected using ICP-MS/MS and orbitrap mass spectrometry employing electrospray ionization operated in the negative mode.

Fluorine detection with ICPMS/MS? A concept of metal-fluorine polyatomic ions

Nor Laili Azua Jamari, Jan Frederik Dohmann, Arne Behrens, Andrea Raab, Eva Krupp, Joerg Feldmann

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Detection of fluorine by inductively-coupled plasma mass spectrometry (ICPMS) is impossible due to the high ionisation potential of fluorine (17 eV). Only small amount of F^+ generated in the argon plasma (15 eV) and high background at m/z 19 due to $^{16}O^{1}H_3^+$ ions restrict the measurement of fluorine with ICPMS. However, through the concept of polyatomic ion interference forms in the plasma, fluorine could be quantified in sub-ppm range. Different metals were evaluated to find the most promising metal-fluorine polyatomic ion ($M-F^+$). Three main characteristics: high bond energy with fluorine, relative low bond energy with oxygen and low second ionisation potential play a role in the formation of $M-F^+$. Barium-fluorine polyatomic ion (BaF^+) at mass-to-charge ratio (m/z) 157 exhibit the highest sensitivity for fluorine detection. Two mechanisms were proposed involving fluorine atom and negative fluorine ion with barium. The method was further developed for fluorine speciation by coupling the high-performance liquid chromatography (HPLC) with ICPMS-triple quad mass spectrometry (ICPMS/MS) and electrospray ionisation mass spectrometry (ESI-MS). Different fluorine compounds exhibit similar sensitivity regardless the structure, which indicates the ICPMS/MS method is fluorine specific detection. ICPMS/MS method able to detect any fluorine compounds which ESI-MS not able to do so. This novel method could be a possible method for analysing unknown fluorine compounds present in environmental and biological samples. Application of this method on the real sample will be presented in the talk.

Sulphur compounds in *Allium* plants and data mining for sulphomics

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Allium vegetables like garlic and shallots are universally consumed because of their flavour, but also as traditional medicine. Both flavour and beneficial health effects are attributed to S-alk(en)yl-L-cysteine sulfoxides, a group of secondary sulphur metabolites.^[1] Further insight into the sulphur metabolism of *Allium* plants is desirable to find connections between fertilisation and these metabolites.

Common analysis methods for these precursor compounds involve the use of species specific standards for quantification and identification. In contrast to this, a coupled HPLC-ICP-MS/ESI-MS system was used in this project to gain elemental, molecular and quantitative information simultaneously.

This poster presents the results of a study examining the influence of different levels of sulphur fertilisation on *Allium* plants. The plants were grown hydroponically with excess sulphate in the nutrition solution. Principle component analysis will be used as a method to find variation in the dataset and could help to find patterns in how the fertilisation influences different metabolites or metabolite groups. An additional focus is on the analysis of the high resolution MS data. The program Dynamic Cluster Analysis (DCA) can be used for the detection of compounds containing heteroatoms like sulphur in HR-MS data without elemental information from an ICP-MS. The manual and automated data mining approaches will be described and compared.

^[1] J. Feldmann, K. Bluemlein, E.M. Krupp, M. Mueller, B.A. Wood, Metallomics study in plants exposed to arsenic, mercury, selenium and sulphur: in Metallomics – the science of biometals, M.A. Zezzi (Ed.), Springer, accepted.

Water-soluble arsenic compounds in the tissue of stranded pilot whales

Savarin Sinaviwat, Johannes Kopp, Andrea Raab, Eva Krupp, Jorg Feldmann

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Arsenic compounds are recognized as potential toxins. It is found that arsenic is widely contaminating the marine environment, with higher concentrations than in terrestrial organisms. Arsenic can be divided in organic arsenic and inorganic arsenic compounds and can be also categorised into water-soluble and lipid-soluble arsenic species. This study investigates the presence of arsenic species in kidney and brain tissue of pilot whales. Samples were extracted to analyse water soluble arsenic and lipid soluble arsenic species. Total water soluble arsenic compounds in the samples were determined by ICP-MS. The compounds were analysed and identified with high performance liquid chromatography separation with simultaneous detection by both inductively coupled plasma mass spectrometry and electrospray ionization mass spectrometry (HPLC-ICPMS/ESI-MS). The investigated compounds are methylarsonic acid (MA), dimethyl arsonate (DMA), trimethylarsine oxide (TMAO), arsenobetaine (AB), arsenosugars, arsenolipids and others unknown species.

A direct online solid phase extraction high performance liquid chromatography inductively coupled plasma mass spectrometry for methylmercury in rice

Parinda Manorut, Eva Krupp, Jörg Feldmann

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Rice is a staple food that easily accumulates trace metals in the grain, including mercury in a neurotoxic form as methylmercury (MeHg) which possibly contribute MeHg exposure risk to rice consumer¹. However, the MeHg content in rice is in trace levels², and the sample preparation for determination can be complicated and time-consuming. A simple and reliable method consisted of online preconcentration or solid phase extraction high performance liquid chromatography cold vapor atomic fluorescence spectrometry (SPE-HPLC-CV-AFS)³ was developed. In this work, the online pre-concentration part consisted of a sulfur-based sorption material for solid phase extraction part is hyphenated to high performance liquid chromatography inductively coupled plasma mass spectrometry (SPE-HPLC-ICP-MS) and improves the LOD and LOQ to 0.08 ng kg⁻¹ and 0.26 ng kg⁻¹ for methylmercury (as Hg) using 40 ml MeHg standard. Then, MeHg concentration in polished (white) and unpolished (brown, red, black) from Thailand was studied and show a difference between types of the grain.

1. Gong, Y. *et al.* Bioaccessibility-corrected risk assessment of urban dietary methylmercury exposure via fish and rice consumption in China. *Sci. Total Environ.* **630**, 222–230 (2018).
2. Brombach, C.-C. *et al.* Methylmercury varies more than one order of magnitude in commercial European rice. *Food Chem.* **214**, 360–365 (2017).
3. Brombach, C.-C. *et al.* Quick and robust method for trace determination of MeHg in rice and rice products without derivatisation. *Anal. Methods* **7**, 8584–8589 (2015).

Chemical Speciation of Antimony using voltammetry in Natural Systems

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The increased use of the metalloid antimony (Sb) has led to many environmental soils and groundwater contamination problems and understanding their biogeochemical cycling is of prime importance to predict potential contamination issues. The speciation of Sb is however often limited to the

differentiation of oxidation states and identification of stable organic species by techniques such as ICP and LC-MS. The detection and identification of more reactive, relatively weak complexes that can play a preponderant role in the transport of these elements are however much less known. One of the aims of this study is to use voltammetry to look at the complexation of antimonite Sb(III) with model ligands. Speciation predictions were obtained using Visual Minteq and using complexation data compiled with Filella and May (2005). Results from pseudopolarographic experiments of Sb(III) on gold electrode gave a wave that was found to be independent of the Sb(III) concentration, but dependent on pH and deposition time as expected. In the case of EDTA and DTPA, two waves were observed at pH 3 and were attributed to the reduction of Sb(III) and of the 1:1 complex, Sb-EDTA or Sb-DTPA. In the former case, stability constant determined experimentally matched those reported by Filella while in the latter case, the stability constant determined experimentally was found 100 times stronger than previously reported. In the case of tartaric acid and glutathione, one wave was observed suggesting the presence strong complex, that cannot be reduced within the deposition potential range tested (-1 V).

Characterization of natural Hg nanoparticles in gas condensate samples: TEM, AF4-ICP-MS, sp-ICP-MS and LA-ICP-MS

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The diversification of feedstock supply in the petroleum industry has led to an increased use of heavy crude oils and gas condensates; many of these more difficult duties contain significant amounts of mercury. The impact of mercury on industrial equipment can be serious, including equipment corrosion¹, catalyst poisoning² and particulate emissions.

These concerns have led to the development of mercury removal units as well as analytical methods for the determination of total mercury; however, the efficiency of the mercury removal units strongly depends on its speciation, which is still debated right now regarding gas condensates³. In this regard, the possible presence of mercury-containing nanoparticles is of concern.

Asymmetric flow field-flow fractionation (AF4) is one of the most promising techniques for particles separation, but it has never been applied to petroleum matrices. We are here presenting a novel method to separate nanoparticles from the dissolved fraction in a “real” gas condensate sample with AF4 using THF as the carrier liquid. The coupling with ICP-MS has highlighted the presence of several elements (Hg but also Fe, P, As, V, Ni and Co) in the nanoparticulate fraction, while TEM imaging confirmed the presence of a large size range (15 to more than 400 nm) of irregularly-shaped nanoparticles. The size of the Hg metallic “core” has been estimated to 60-80 nm using Single Particle-ICP-MS. Finally, LA-ICP-MS imaging showed that Hg particles can sometimes coexist with other elements such as S or As.

Selenium nanoparticles produced by the fungus *Aureobasidium pullulans*: Separation, detection and sizing by asymmetric flow field flow fractionation (AF4) with online inductively coupled plasma mass spectrometry (ICPMS).

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Selenium is an economically important but rarely occurring element, used in manufacture of semi-conductors, photocells, glass, and pigments. Its low natural abundance has heightened supply security fears leading to the exploration of environmentally sustainable ways for its production. Here, we report the detection and sizing of selenium nanoparticles obtained from a fungus, *Aureobasidium pullulans* grown in a liquid nutrient medium amended with 1 mM Na₂SeO₃ for 10 days at 25°C. We developed an optimised AF4 separation method in aqueous mode (0.2% Novachem as eluent, and an exponential elution crossflow of 4 mL min⁻¹), with online coupling to ICPMS for element specific detection of selenium on m/z 78. Integration of the MALS peak with the Postnova® AF2000 MT software resulted to two size distributions with radii of gyration (R_g) ranging from 55-65 nm, and 66-80 nm; with a good data fit across all 21 angles using a spherical model. This positively correlates with single particle ICPMS data which showed a narrow size distribution with a median particle diameter of 70 nm. Our study thus highlights the possibility of a sustainable method for the biological sequestering of selenium from the solution in the form of pure elemental selenium. It also demonstrates the robustness of hyphenated ICPMS techniques for the characterization of such natural nanoparticles in complex biological media.

CACO-2 IN VITRO MODEL OF HUMAN GASTROINTESTINAL TRACT FOR STUDYING ABSORPTION OF TITANIUM DIOXIDE NANOPARTICLES FROM SEAFOOD SAMPLES

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Titanium dioxide nanoparticles (TiO₂ NPs) are used in industry, as an inorganic UV filter in sunscreens and personal care products, and also as a food additive (E171). The huge increase in the use of nano-based products, mainly metallic NPs, implies the presence of nanomaterials in the environment, and hence, the unintentional human ingestion through water or foods (gastrointestinal, GI, tract is the main pathway of NPs intake in humans) [1].

The presence of TiO₂ NPs in seafood samples was firstly established using an ultrasound assisted enzymatic hydrolysis procedure and sp-ICP-MS analysis. Several clams, cockles, mussels, razor shells, oysters and variegated scallops, which contain TiO₂ NPs, were subjected to an *in vitro* digestion process [2]. The selected GI model was Caco-2 cells because of the development of membrane transporters that are responsible for the uptake of chemicals [3]. Parameters as trans-epithelial electrical resistance (TEER) and permeability of Lucifer Yellow were studied for establishing monolayer integrity. TiO₂ NPs transport and apparent permeability through GI-epithelial barrier (0-2 hours) have been assessed by sp-ICP-MS.

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Alkaline Digestion as a Sample Pre-Treatment for Titanium Dioxide Nanoparticles Extracted from Surimi and Seafood Samples

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The remarkably increase in the use of nanoparticles (NPs) in many applications implies a need to evaluate their impact on human health and environment [1]. Titanium dioxide nanoparticles (TiO₂ NPs) are hugely used in industry, as an inorganic UV filter in sunscreens and personal care products, and also as a food additive (E171) [2].

TiO₂ NPs in surimi and seafood samples have been studied using an alkaline digestion. Some alkaline extractions have been reported in the literature [3] but there are not data regarding TiO₂ NPs extraction from food matrices. Optimum extraction conditions imply 2 mL of TMAH 10% (v/v) and sonication (ultrasound water-bath) for 2 hours. Repeatability, limit of detection and analytical recovery with TiO₂ NPs standards of different sizes were studied. TiO₂ NPs presence in different types of clams, cockles, mussels, razor shells, oysters and variegated scallops and surimi samples (11 samples) have been previously confirmed by an ultrasound assisted enzymatic extraction and sp-ICP-MS analysis. They also were analyzed for total titanium (ICP-MS after acid digestion).

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Assessment of Zinc Oxide Nanoparticles in Textiles by Single Particle ICP-MS

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The modification of textile fibres with nanoparticles (NPs) offers to customers clothing with innovative properties. Thus, metallic NPs provide protection against ultraviolet light (TiO₂ NPs, ZnO NPs), water resistance (SiO₂ NPs, ZnO NPs), antimicrobial activity (Ag NPs, TiO₂ NPs, MgO NPs, CaO NPs, CuO NPs), and flame retardant characteristics (ZnO NPs, SiO₂ NPs, TiO₂ NPs) [1-3]. However, the release of these NPs from textiles can occur during laundering, and they can therefore reach wastewater treatment plants. NPs can be accumulated in sewage sludge, by-product which is commonly used as a fertilizer. Otherwise, NPs can migrate through sweat and can penetrate into the skin, being a risk for human health. The development of methods for assessing NPs in textiles is therefore necessary.

In the current communication, a simple extraction pre-treatment (sonication with water as a solvent) has been used for isolating ZnO NPs from textiles before sp-ICP-MS assessment. The whole method has been optimized and validated, and it has been applied to several clothing.

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The effect of sample drying on the operational speciation of potentially toxic elements in sediment using the modified BCR sequential extraction procedure

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The BCR sequential extraction procedure is widely used to determine the operational speciation of potentially toxic elements in soils and sediments. When applying the approach, a dichotomy exists between maintaining a sample's intrinsic speciation, and pre-treating the sample to make it stable and amenable to standard laboratory operations such as sieving, cone-and-quartering etc. Sample drying in particular has been reported to cause redistribution of analytes to more readily extractable forms. A common compromise is to air-dry samples at < 25 °C. However, many studies report findings based on extraction of sediments subjected to much harsher pre-treatment regimes.

In order to assess the effects of sample drying on the operational speciation of PTE in sediment, the modified BCR sequential procedure was applied to samples obtained from the catchment of the River Derwent (an area impacted by historical mining and smelting) following various pre-treatment regimes. Samples were analysed as received, air-dried, freeze-dried, and following oven drying at 60°C and 110°C. Extracts were analysed for As, Cd, Cu, Fe, Mn Ni, Pb, U and Zn using an Agilent 7700x ICP-MS instrument.

Different changes in operational speciation occurred for different analytes. Freeze-drying increased the proportion of Pb associated with the exchangeable and acid soluble fraction. In general the proportion of Cd, Cu and Zn associated with the oxidisable phase increased on drying up to 60°C but decreased at 110°C. Manganese and Fe were less affected by drying, whereas the behaviour of As differed between samples.

Seasonal investigation of potentially toxic elements in the vicinity of a major e-waste recycling site in Lagos, Nigeria

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Alaba International Market is one of the oldest and largest e-waste recycling site in Nigeria. The e-wastes are recycled crudely with minimal regard to environmental impact. The influence of these activities on the concentrations of potentially toxic element in and around the site is of concern and requires investigation.

Soil samples from the vicinity of the Alaba e-waste recycling site in Lagos were collected during wet, intermediate and dry seasons for two years. They were dried, sieved and subjected to microwave-assisted aqua regia digestion. The digests were analysed for Cu, Cr, Fe, Mn, Pb and Zn and using an Agilent 7700x ICP-MS instrument.

The results of the pseudototal analysis showed that the concentrations of the potentially toxic elements were higher than permissible limits recommend by the Nigerian Federal Environmental Protection

Agency Act (1991). Higher concentrations were generally recorded in the dry seasons. Levels were dependent upon proximity to the recycling site and activities carried out there.

Comparison of on-site field measured inorganic arsenic in rice with laboratory measurements using a field deployable method: method validation

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A commercial arsenic field kit designed to measure inorganic arsenic (iAs) in water was modified into a field deployable method (FDM) to measure iAs in rice. While the method had been validated to give precise and accurate results in the laboratory, its on-site field performance has not been evaluated. This study was designed to test the method on-site in Malawi in order to evaluate its accuracy and precision for the determination of iAs on-site by comparing on-site iAs results with those obtained using validated reference method; and to obtain original data on iAs in Malawian rice. The method was validated by using the established laboratory-based HPLC-ICP-MS/MS. Statistical tests indicated there were no significant differences between on-site and laboratory iAs results determined using the FDM ($p=0.263$, $\alpha=0.05$) and between on-site results and results determined using HPLC-ICP-MS/MS ($p=0.299$, $\alpha=0.05$). Furthermore, both FDM and HPLC-ICP-MS/MS analyses gave low false positive (9.1% and 0%) and low false negative (3% and 0%) results for samples with low (<100 µg/kg) and high (>100 µg/kg) iAs values respectively. The findings strongly indicate that results obtained in the field using FDM are reproducible (high precision) with reference methods in a laboratory. This method also allows quick (within 1 hour) and efficient screening of iAs concentrations in rice on-site.

Effect of fertiliser choice on elemental composition of bere barley crops

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Elemental/isotopic compositions of skeletal remains can be used to investigate past diets and geographical origins of people and animals. Fertilisation of crops has been shown to cause difficulties for such studies, as it can influence the composition of the crops to mimic other foods, such as meat (in case of $\delta^{15}\text{N}$). Particularly the effects of seaweed fertilisation on crops require further research, with relevance not only to archaeology, but also to nutritional sciences and food chemistry.

A fertilisation trial was undertaken on Orkney, where selected plots of bere barley were fertilised with seaweed (10 t/ac and 20 t/ac), NPK fertiliser (50 kg N/ha) or left unfertilised. Yield, and grain, husk and straw elemental compositions were determined by MP-AES (Mg, K, Ca, Mn, Fe, Cu, Zn and Sr) and ICP-MS (B, V, Cr, Co, Ni, Zn, As, Br, Sr, Mo, Cd and Pb). Concentrations of B, Sr, As, Mn and Cd were elevated in at least some parts of the plants in the seaweed-fertilised crops compared to unfertilised samples; however, Mo and Br appear to be less concentrated in seaweed- and NPK-fertilised crops than in unfertilised crops. This is likely attributable to the effects of soil chemistry. Principal component analysis of these results showed a clear separation of the three different fertilisation treatments (seaweed, NPK and no fertiliser) on the basis of As, Mo, Mn, Sr, B and Cd concentrations (PC1 53 %, PC 2 22 %), indicating that seaweed fertilisation significantly impacts the chemical composition of crops.

Elemental analysis of maize impacted by the cement industry in North-Central Nigeria

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Maize is one of the most significant cereal grains in the world and is a foremost staple diet of many Nigerians and Africans eaten by people with variable socio-economic circumstances and food inclinations¹. Maize also serves as feed for domestic animals and its production has surpassed the traditional cereals like millet and sorghum. It is hypothesized that it could also pose a risk to human health and environmental harm depending on the farming location.

Plants serve as the initial step of a metal's route to entering the food chain of humans and animals from the soil². Therefore, the concentration of nutritionally, toxicologically and environmentally-important major, minor and trace elements in the edible parts of a plant denote accessible load that could enter the food chain through plants².

In this study, maize grains were sampled from five farmlands including control during the maize growth season in an area of active cement manufacturing in North-Central Nigeria. We have analysed a range of essential and toxic elements using MP-AES and ICP-MS to assess the impact of the cement factory in the vicinity, and in a control site. The results are aimed at generating data for impact studies which could be useful for improving food quality.

Keywords: Maize; Cement factory; Essential elements; Toxic elements; Trace Elements

¹ https://www.afdb.org/fileadmin/uploads/afdb/Documents/Events/DakAgri2015/Cereal_Crops-Rice_Maize_Millet_Sorghum_Wheat.pdf

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Total elemental analysis of food samples with ICP-OES and ICP-MS

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The measurement of toxic, essential and nutritional elements in food has, through regulatory drivers and today's health-conscious consumers, become a routine part of food quality monitoring. Alongside regulatory compliance, it is necessary to monitor potentially toxic contaminants that could enter the food chain via a series of pathways including, but not limited to, industrial pollution or environmental contamination. For these reasons, it is essential to have a simple, robust, multi-elemental analysis method for detecting and quantifying major and minor concentrations of elements in food.

ICP-OES and ICP-MS are sensitive and rapid techniques with wide linear dynamic range and as such are ideal tools for the analysis of trace and major analytes in food together in one analytical run. The principal challenge for trace elemental ICP-based techniques are interferences that stem from the complex food matrix, the reagents used to prepare the sample and the plasma source. This paper reviews different strategies, including collision/reaction cell (CRC) approaches in triple quadrupole ICP-MS, for the accurate analysis of trace elements in food sample matrices. The accuracy of the techniques reviewed is demonstrated by the analysis of food based certified materials following microwave digestion.

Overview of applications for ICP-MS in Food Industry services.

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Campden BRI specialises in providing analytical services to the Food and Drink industries, encompassing testing of food, beverages, food additives, pet and animal feeds, packaging and migration from packaging; for both toxic and nutritional elements. Traditionally applied for low-level toxic element screening, we employ ICP-MS to analyse abundant nutritional elements including sodium and equivalent salt content using sensitivity reduction strategies. Simultaneous measurement of major and trace elements also has application in Food Authenticity and identification of unknown substances for Flavour and Taint investigations and Foreign Body identification. ICP-MS is also applied to speciation analysis particularly for inorganic arsenic and selenium.
