I001

MOF-BASED POTENTIOMETRIC PVC MEMBRANE SENSORS FOR THE DETERMINATION OF ACTIVE DRUG SUBSTANCES. Serkan Tekce,¹ Gulsah Saydan Kanberoglu,¹ Fatih Coldur² and Yaver Subasi,¹ Van Yuzuncu Yıl University, Department of Chemistry, Turkey, ²Erzincan Binali Yildirim University, Department of Chemistry, Turkey, (serkan.tekce@saglik.gov.tr, gskanberoglu@yyu.edu.tr, fcoldur@erzincan.edu.tr, yaversubasi@hotmail.com)

Metal-organic lattice structures are formed by the copolymerization of organic ligands and transition metals, where the organic bridge-inorganic metal ion pair of these materials ensures the formation of a perfect mixed matrix membrane structure [1]. Imipramine-selective and maprotiline-selective potentiometric sensors were developed by using Mil-53(Al), one of the metalorganic lattice structures, as an ionophore in the electrode structure. MOF-based PVC membrane electrode was successfully employed to determine the imipramine hydrochloride and maprotiline hydrochloride contents of some drugs used in the treatment of depression. When maprotiline hydrochloride is used in overdose, very serious side effects such as shock, heart failure, loss of muscle coordination, hyperactivity, irregular heartbeat, difficulty breathing and even shortness of breath can occur [2]. Similarly, side effects such as insomnia, crying spells, personality change and tachycardia can result from overdose with imipramine hydrochloride. MOF-based potentiometric sensors were developed for the determination of maprotiline hydrochloride and imipramine hydrochloride. The linear concentration ranges for maprotiline and imipramine hydrochloride were found to be respectively 1.0×10^{-9} M -1.0×10^{-2} M, 1.0×10^{-7} M -1.0×10^{-1} M with a slope of 59.1 mV/decade and 57.7 mV/decade and a detection limit of respectively 9.0×10^{-10} 10 M and 5.0×10⁻⁸ M in the pH range 2-9.5 and 3.7-8.4. The applications have been carried out in pharmaceutical tablets.

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I002

BATTERY RESEARCH USING VIBRATIONAL SPECTROSCOPY. Sergey Shilov, Bruker Optics, 40 Manning Rd., Billerica, MA, USA. (Sergey.Shilov@bruker.com)

The combination of electrochemistry with infrared and Raman spectroscopy is a powerful tool for studying electrochemical reactions, battery research, and failure analysis. Each molecule has a unique infrared and Raman signature providing great specificity in the analysis of the molecular change during electrochemical reactions.

An overview of the spectroelectrochemical tools, optimization of the setup, and details of communication between the potentiostat and the spectrometer will be presented. Examples of applications will include electro-oxidation of metal-organic complexes, carbon analysis in flexible electrodes, and chemical mapping of the solid electrolyte interface of lithium metal batteries.

I003 P

NOVEL SEPARATIONS USING A MODIFIED WATER STATIONARY PHASE IN SFC **Emmanuel Nai**, and Kevin Thurbide, Department of Chemistry, University of Calgary, 2500 University Drive NW, Calgary, Alberta, T2N 1N4, Canada. (<u>emmanuel.nai@ucalgary.ca</u>)

Stationary phase development is an important area of research in supercritical fluid chromatography (SFC) due to the impact that it can have on separation selectivity and efficiency. Water has interesting properties in this regard, such as its non-toxicity, low cost, strong polarity, and available purity, among other things. Even so, water has predominantly only been explored as a mobile phase additive in SFC. Recently, we introduced a novel method of using water as a stationary phase in capillary column SFC. The method provided excellent peak shape, reproducibility, and flame ionization detector compatibility for a variety of polar solutes using only carbon dioxide as the mobile phase, without modifier present. Here, we present new modes of operation with this method that allow a wider variety of polar ionizable analytes to be separated in this way while maintaining good efficiency and detector compatibility. Operating conditions, method characteristics, and some example separations will be presented and discussed.

I004

PROGRESSIVE ANALYSIS OF NANOMATERIALS IN ENVIRONMENTAL MATRICES – SELECTED ASPECTS FROM SAMPLING TO DATA EVALUATION. **Petra Krystek**, Vrije Universiteit (VU), Amsterdam, Dep. Environment & Health, De Boelelaan 1108, Amsterdam, 1081 HZ, The Netherlands; Andreas Wimmer, Technical University Munich, Germany; Rob Ritsema, RR Quality Consultancy, Amersfoort, the Netherlands; Carsten Engelhard, University of Siegen, Germany. (petra.krystek@vu.nl)

Engineered, inorganic nanomaterials are studied in environmental compartments for exposure and risk assessments while possible bottlenecks are the related validations of complete methods and quality criteria. Workflows often starts with sample pretreatments in the laboratory followed by instrumental analysis for the identification and/or detection of nanoparticles. But it is obvious that especially for the ultra-trace analysis of nanomaterials, the sampling is of the utmost importance in analytical workflows. The sampling is often underexposed and possible errors at these steps influence the validity of results [1,2]. Several examples will be discussed during the presentation. For a progressive analysis of nanomaterials, more aspects become relevant and the concept on the development of nanotechnology products or nanotechnology applications is divided into four generations. This concept is based on an increasing level of sophistication and complexity of systems at the nanoscale. Currently, analytical nano-science is in transition from the first generation of passive nanomaterials to the second generation of active nanomaterials. This transition of related characterization methods in plasma spectrochemistry is challenging. By a case study for active nanomaterials, the current state of analytical methods and the knowledge on risk assessments of future generations is explored and the results will be presented [3].

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ANALYTICAL METHOD DEVELOPMENT AND VALIDATION FOR ESTIMATION RIVAROXABAN IN ITS PHARMACEUTICAL DOSAGE FORM BY LC-MS/MS. Lalitkumar Pankajkumar Patel, Saraswati Institute of Pharmaceutical Sciences Near Anjali In Hotel, Ahmedabad-Himatnagar Highway, At & Post: Dhanap, Ta & Dist: Gandhinagar – 382355, Gujarat, India. (lpatel22@gmail.com)

A LC-MS/MS method was developed and validated for estimation of Rivaroxaban in Tablet Dosage form. The mass spectra were recorded in electron spray ionization (ESI) in positive mode of detection. Nitrogen was used as curtain gas. The ion source condition were set as follows: Temperature, 400°C; Curtain Gas, 20psi; Ion Spray Voltage, 5200V; Ion Source Gas (GS1), 60psi and Ion Source Gas (GS2), 60psi. The scan condition was set as follows: Scan type, MRM; Declustering Potential, 32V; Focusing Potential. From the mass spectra it is found that, rivaroxaban molecular mass is found around m/z 436.300 Da and its fragmentation molecular mass is found around m/z 436.300 Da and its fragmentation molecular mass is found around m/z 436.300 Da and its fragmentation molecular mass is found around m/z 436.300 Da and its fragmentation molecular mass is found around m/z 436.300 Da and its fragmentation molecular mass is found around m/z 436.300 Da and its fragmentation molecular mass is found around m/z 436.300 Da and its fragmentation molecular mass is found around m/z 260.400 Da. The developed method was validated as per ICH guidelines. The linearity was investigated in the range of 0.5-1.5µg/mL (r2 = 0.999) for Rivaroxaban. The LOD were 0.005µg/ml for Rivaroxaban, respectively. The LOQ were 0.016µg/mL for Rivaroxaban, respectively. This method was found to be simple, accurate, robust and reproducible. A new LC-MS/MS method has been developed for estimation of Rivaroxaban in tablet dosage form was rapid, accurate, precise, economic and easy to perform.

I006

SUNLIGHT DRIVEN PHOTOCATALYTIC DEGRADATION OF TETRACYCLINE ANTIBIOTIC BY BIOGENIC SYNTHESISED MNV₂O₆/BIVO₄ HETEROJUNCTION: ROUTES OF DETERIORATION AND MECHANICAL PERCEPTIONS. **Sandeep Kaushal** Department of Chemistry, Sri Guru Granth Sahib World University, Fatehgarh Sahib, Punjab, India. (kaushalsandeep33@gmail.com)

Tetracycline (TC) is a commonly used antibiotic in many countries due to its low cost and high efficacy. Particular antibiotics, on the other hand, can be hazardous to living beings due to metal ion complexation, which can cause teratogenicity and carcinogenicity. In this study, MnV2O6/BiVO4 heterojunction was constructed using *carica papaya* extract as a reductant and capping agent in a green synthesis technique that was both environmentally friendly and cost effective. The efficacy of the as-synthesized MnV₂O₆/BiVO₄ heterojunction as a photocatalyst for TC antibiotic degradation was investigated utilizing a variety of sophisticated methodologies. The MnV₂O₆/BiVO₄ heterojunction exhibited exceptional efficiency for degradation of TC antibiotic (95% removal in 50 min), with apparent rate constant of 0.062 min⁻¹. Photocatalytic activities of MnV₂O₆/BiVO₄ heterojunctions were investigated in degradation experiments by altering parameters such as contact time, catalyst dose, and solution pH. Radical scavenging investigations revealed that reactive species play a role in the breakdown of antibiotics and dyes. In photocatalytic breakdown, the OH radical is crucial. Moreover, experiments using liquid chromatography-mass spectrometry (LC-MS) were used to predict a feasible mechanism for TC degradation.

I007

ESTABLISHING THE MECHANISM FOR IONIC LIQUID CATALYZED CARBAMATION OF AMINES USING SPECTROSCOPIC TECHNIQUES. Khushboo¹, Ashutosh Sharma¹, Jasminder Singh^{2,3}, **Rahul Badru¹** and Swami Premanand Mahavidyalaya². ¹Department of Chemistry, Sri Guru Granth Sahib World University, Fatehgarh Sahib, Punjab, India-140406; ²Department of Chemistry, IIT Ropar, Punjab, India-140406. (*rahulbadru@gmail.com*)

Reaction of amines with dimethylcarbonate (DMC) lead to the generation of carbamates as well as *N*-methylated products [1]. A number of reagents have been explored for achieving selectivity towards one of these products in the reaction. In this work, we investigated the reaction of amines with DMC in the presence of 1-alkyl-3-Methylimidazolium [MIm] derived ionic liquids (ILs). The reaction afforded carbamates with 100% selectivity, as judged by in-situ GC analysis of the reaction mixture. It was demonstrated that the IL served as the bifunctional catalyst activating both the amine substrate and DMC to interact with each other. The proton flanked by two nitrogens in the imidazolium cationic part of the IL interacts and increases the nucleophilicity of the carbonyl functionality of DMC [2], thereby increases the chances of attack by the amine substrate. The proposed interactions have been well established by signal shifting in the NMR spectrum and supported by DFT studies.

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I008

CONVICINE AND LEVODOPA FROM FABA BEAN USING FLOW INJECTION ANALYSIS-HIGH FIELD ASYMMETRIC WAVEFORM ION MOBILITY MASS SPECTROMETRY. **Haixia Zhang**. The University of Winnipeg, Department of Chemistry, 599 Portage Ave., Winnipeg, MB R3B 2G3, Canada. (<u>h.zhang@uwinnipeg.ca</u>)

Faba bean, due to its high content of protein, micronutrients and dietary fiber, is a promising crop for the food industry. However, the presence of vicine and convicine (v/c), which could cause favism in people with glucose-6-phosphate dehydrogenase deficiency, has limited faba bean consumption. Levodopa (L-DOPA) is a nutraceutical compound present in faba bean seeds, which is the major ingredient in treating Parkinson's disease. We developed a rapid (1-min) and robust approach for v/c and L-DOPA quantification, using flow injection analysis in combination with high-field asymmetric waveform ion mobility spectrometry mass spectrometry (FIA-FAIMS MS). This method was validated on two FAIMS devices and matching mass spectrometers. Good measurement accuracy and reproducibility were observed from both platforms. Compounds were extracted from faba bean seed powder with 1% formic acid, and diluted $50 \times$ in 10% acetonitrile before analysis. FIA-FAIMS was performed on a triple quadrupole mass spectrometer in selected reaction monitoring mode (SRM). This approach was then applied to quantify these compounds in a large number of faba bean genotypes and faba-based food ingredients. To the author's knowledge, this is the first reliable quantification approach for v/c and L-DOPA.

LABEL-FREE PATHOGEN DETECTION USING BROAD SPECTRU BIORECOGNITION ELEMENT; Jennifer F. McLeod, R. Stephen Brown, Zhe She; Queen's University, Department of Chemistry, Kingston, ON K7L 3N6, Canada. (j.mcleod@queensu.ca)

The 2021 UN World Water Development Report highlights the undervaluation of water, impacting water accessibility and climate action. Despite Canada's relative wealth, there is a serious water crisis amongst many First Nations communities, where several short- and long-term drinking water advisories are in effect. The SARS-CoV-2 pandemic highlighted the necessity for access to clean water to guarantee appropriate sanitation. Ensuring the potability of water for basic human needs requires time consuming and costly analytical methods, highly qualified personnel, and equipment. Point-of-care (POC) devices eliminate the need for highly qualified persons and equipment, allowing tests to be performed on site.

The demand for rapid detection with high sensitivity and low cost has increased attention toward electrochemical biosensors. A biosensor contains three key parts: a biorecognition element (BRE), a sensor chip, and a linker molecule which connects the two. Using a gold sensor chip, whole cell pathogen detection by toll-like receptor BREs is demonstrated.^{1,2} The result demonstrates the versatility in the biosensor scaffold, with implications for multi-analyte detection.

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 Singh, I. *et al. Chem Commun* **2021**, 57 (68), 8421-8424

I010

GREEN SYNTHESIS AND BIOLOGICAL SCREENING OF SILVER NANOPARTICLES FROM AQUEOUS EXTRACT OF *EUPHORBIA PROSTRATA*. **Vipan Kumar^{1*}**, Prabhakar Kumar Verma², Anupama Setia¹ ¹JCDM College of Pharmacy, Sirsa-125055, India; ²Department of Pharmaceutical Sciences, Maharshi Dayanand University, Rohtak-124001, India (<u>vk.kamboj123@gmail.com</u>)

Euphorbia prostrata aqueous extract was used to synthesize silver nanoparticles (AgNPs), characterized by UV-Visible spectroscopy, XRD, and SEM, and screened for their antimicrobial and anticancer (against MCF-7 breast cancer cell line) activity. The silver ions reduced to AgNPs in presence of aqueous extract and change in color was observed due to surface plasmon vibrations in AgNPs [1]. UV-Vis spectroscopic analysis gives peaks on 438 nm indicates the formation of AgNPs. XRD analysis showed intense peaks at 20 values of 38.62° , 44.82° and 65.08° corresponding to (111), (200), and (220) Bragg's reflection based on the fcc structure of AgNPs. SEM analysis of AgNPs showed spherical and truncated triangles morphologies ranging from 25 to 80 nm. Antimicrobial activity screened against gram positive and gram negative bacterial strains produced the dose-dependent antimicrobial effect. The IC₅₀ value of prepared AgNPs showed significant improvement in anticancer potential compared to silver nitrate. The biologically synthesized *Euphorbia prostrata* AgNPs can be used in the medical field for their efficient antimicrobial, and anticancer potential after undertaking the proper clinical trial.

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I011

DEVELOPMENT AND VALIDATION OF STABILITY INDICATING RP-HPLC METHOD FOR ESTIMATION OF AMINOCAPROIC ACID IN TABLET DOSAGE FORM. **Paresh U. Patel**, Ganpat University-Shree S. K. Patel College of Pharmaceutical Education and Research, Ganpat Vidyanagar – 384012, Mehsana, Gujarat, India. (paresh.patel@ganpatuniversity.ac.in)

A stability indicating RP-HPLC method was developed and validated for estimation of aminocaproic acid in tablet dosage form. The chromatographic separation was achieved on InertsilC8-3 (250 mm × 4.6 mm, 5µm) column using mobile phase phosphate buffer:methanol (65:35 v/v) adjust buffer pH 2.2 with orthophosphoric acid solution at a flow rate of 0.7 mL/minute. The detection was carried out at 210 nm. The developed method was validated for system suitability, precision, linearity, accuracy, specificity, robustness as per ICH guidelines. Linearity of the proposed method was found in the concentration range of 250 µg/mL to 750 µg/mL with regression coefficient of 0.9990. The recovery was found to be 99.40 ± 0.75. LOD was found to be 5.83 µg/mL and LOQ was found to be 17.66 µg/mL. Stability studies were conducted by adopting the proposed method to assess the stability of standard and sample solution under acid, alkaline, peroxide, thermal and photolytic condition. From the experimental studies it can be concluded that proposed method can be applied as stability indicating for estimation of aminocaproic acid in pharmaceutical dosage form without interference of excipients and degradation products.

I012 P

DESIGN AND DEVELOPMENT OF ELECTROCHEMICAL IMMUNOSENSOR FOR BRAIN-RELATED PROTEIN. **Meaghan Tabobondung**, William Wallace, Sanela Martic. Trent University, 1600 West Bank Drive, Peterborough, ON K9L 0G2, Canada. (sanelamartic@trentu.ca)

The process of diagnosis for neurodegenerative diseases relies on the onset of symptoms. However, there is the possibility of detecting these diseases, such as amyotrophic lateral sclerosis (ALS), before progression, which would allow for more treatment. ALS targets the motor system and eventually leads to total muscle paralysis. Early detection of ALS can be achieved by detecting biomarkers associated with the disease, one of which is the TDP-43 protein. Using electrochemistry, miniaturized and portable biosensors which provide rapid response, are ideal for the point of care applications. Towards this goal, we developed a label-free electrochemical impedance spectroscopy (EIS) biosensor. The signal output was the charge transfer resistance (Rct) of the antibody-surface before and after protein exposure. The changes in the Rct values were directly related to the amount of analyte. The fabricated sensor was a TDP-43 antibody-modified gold disk working electrode. After surface modification, the immunosensor optimization was carried out by using several commercial TDP-43 antibodies at various concentrations. The significant changes in Rct values were observed above 100 nM concentration of the TDP-43 protein. TDP-43, but can be easily extended for detection of other disease-related biomarkers.

DESIGN AND DEVELOPMENT OF CHEMICAL SENSORS AND BIOSENSORS FOR FORENSIC APPLICATIONS. **Sanela Martic**, Department of Forensic Science, Trent University, 1600 West Bank Drive, Peterborough, ON K9L 0G2, Canada. (sanelamartic@trentu.ca)

Forensic evidence such as drugs, metabolites, gunshot residue, among others, plays an integral role in forensic cases and their outcomes. Commonly, the presumptive testing is carried out prior to confirmatory testing in the laboratory and by using specialized equipment. In this talk, common presumptive and confirmatory testing methods will be described, such as colour-based methods, spectroscopy, fluorescence, mass spectrometry, and electrochemistry. The standard testing procedures in forensic chemistry will be compared to alternative new methods based on nanomaterials or electrochemistry for development of chemical sensors and biosensors for forensic applications.

I014

FAST GC-MS METHOD FOR IDENTIFICATION AND QUANTIFICATION OF TERPENES FROM *CANNABIS* SAMPLES. Abdul Qudeer Ahmed¹, Yoonjo Park¹, David Noshad², and **Paul C.H. Li¹**. ¹Department of Chemistry, Simon Fraser University, 8888 University Drive, Burnaby, British Columbia, Canada; ²Medcan Biotechnologies Inc., Vancouver, BC, Canada. (paulli@sfu.ca)

Of all the natural products found in plants, over 50 000 terpenoids have been discovered, making them the largest class of compounds identified [1]. It has been reported that around 200 terpenes exist in *Cannabis* [2]. In *Cannabis*, many terpenes are bioactive and they play a vital role in anticancer, anti-fungal, anti-viral, anti-inflammatory, and anti-parasitic properties [3, 4]. So far, terpenes have been mostly determined by GC-mass spectrometry (GC-MS) [2, 5] and GC-flame ionization detection (GC-FID) (4). However, there has been little research directly investigating the rapid method for terpene detection in *Cannabis* samples that includes *C. sativa*, *C. indica*, *Cannabis* 5-CW, Rock Star, and Super Silver. The aim of this study was to chemically analyze different *Cannabis* samples and develop a user-friendly method to detect important bioactive terpenes. The study presented here is one of the first investigations to utilize GC-MS to systematically quantify *Cannabis*-derived terpenes, such as α -pinene, β -pinene, myrcene, limonene, β -caryophyllene, and α -humulene.

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ELECTROCHEMISTRY OF NEURONAL PROTEINS AND PEPTIDES. Sanela Martic, Department of Forensic Science, Trent University, 1600 West Bank Drive, Peterborough, ON K9L 0G2, Canada. (sanelamartic@trentu.ca)

Tau is a neuronal protein and one of the biomarkers of neurodegenerative diseases, such as Alzheimer's disease. Tau protein undergoes post-translational modifications, aggregation, and is a viable drug target. In addition, this protein is a vital biomarker in biological fluids towards early detection of neurodegenerative diseases. We reported on using electrochemical impedance spectroscopy and cyclic voltammetry for detection of protein-protein interactions, protein-ligand interactions, enzymatic catalysis and enzyme inhibition while focusing on tau protein and its biochemistry. For example, tau-tau, tau-ferritin, tau-transferrin protein interactions were monitored, and protein kinase-catalyzed phosphorylation of tau protein was detected. The phosphorylation inhibitors, such as antibodies, were screened for their efficacies, and electroimmunosensor developed for tau detection. The methodology was also applied for monitoring metal ion coordination by tau peptides. More recently, we expanded the electrochemical strategies towards ALS-related protein. Bioelectrochemical methodologies were used to gain insight into various facets of protein biochemistry, and represent promising tools in neuroscience research.

I016

QUANTIFICATION OF SURFACE TOPOGRAPHY OF TREATMENT WETLAND BIOFILM. Anbareen J. Farooq¹, **Mhari Chamberlain**¹, Arman Poonja¹, Dr. Kevin G. Mumford², Dr. Kela P. Weber¹, ¹Environmental Sciences Group, Department of Chemistry and Chemical Engineering, Royal Military College of Canada, Kingston, ON K7K 7B4, Canada; ²Department of Civil Engineering, Queen's University, Kingston, Ontario K7L 3N6, Canada. (mhari.chamberlain@rmc-cmr.ca)

Biofilms are highly heterogeneous and hydrated structures, which can be found in a variety of both natural and man-made environments. Biofilms house diverse microbial communities within a self-made matrix of extracellular polymeric substances. These microorganisms are responsible for the majority of wastewater constituent degradation and transformation in treatment wetlands (TWs), a nature-based wastewater treatment technology. In the TW field, many studies have focused on investigating the activity, function, and structural diversity of biofilm; however, few have focused on characterizing their morphology. This study focuses on imaging the morphology of TW biofilm using a scanning electron microscope with a wet stage (Wet-SEM). Wet-SEM imaging prevents sample dehydration by adjusting temperature and humidity conditions. As biofilm is composed of over 90% water, Wet-SEM maintains the hydrated morphology that is lost under conventional SEM methods. Biofilm samples (detached and on gravel surfaces) from both aerated and non-aerated TWs were harvested after two years of operation and imaged with Wet-SEM. This imaging technique provided a 3D visualization of a porous and hydrated structure of the TW biofilm surface, which has not been previously seen.

A SIMPLE LOW-COST 3D PRINTED SHEATH FLOW CUVETTE FOR SENSITIVE FLUORESCENCE DETECTION IN CAPILLARY ELECTROPHORESIS. Cameron D. Skinner, Concordia University, Department of Chemistry & Biochemistry, Montreal, QC, Canada. (Cameron.Skinner@concordia.ca)

Sheath flow cuvettes (SFC) offer some of the lowest detection limits for capillary electrophoresis because detection actually occurs post-column and in a flowing stream of refractive index matching sheath buffer. This eliminates scatter from the capillary/air and sample/capillary interfaces a major contributor to the blank noise. Unfortunately, many SFC are precision detection cells that require expensive laser excitation sources, optics and precision alignment. In this work inexpensive 3D printing, 488 nm laser diodes and fiber optic excitation are used to simplify the production of a SFC. Precise alignment of the fiber optic with the capillary effluent is accomplished using a simple adjustable flexure arm that holds the fiber optic. A beam trap was incorporated into the SFC to prevent absorption of the excitation radiation by the SFC wall. Without it, thermal convective flows in the channel destroyed the all-important laminar flow. With the prototype SFC we have achieved detection limits of 30 pM Fluorescein in 50 mM Borate buffer but, the system and design are still being optimized and characterized.

I018

ELETROCHEMICAL IMPEDANCE SPECTROSCOPY AND CYCLIC VOLTAMMETRY FOR THE CHARACTERIZATION OF SUPERCAPACITORS. **Dominik P.J. Barz**, Department of Chemical Engineering, 19 Division Street, Kingston, ON K7L 3N6, Canada. (dominik.barz@queensu.ca)

Supercapacitors can provide high specific electric power with outstanding cyclic stability and efficiency. They are widely used in various applications ranging from regenerative braking in (hybrid) electric vehicles to uninterruptible power supplies. Supercapacitors can be divided into the three basic categories: Electrical Double Layer capacitors (EDLC), pseudo-capacitors and hybrid supercapacitors according to the energy storage mechanisms. In this talk, we discuss the application of Electrochemical Impedance Spectroscopy and Cyclic Voltammetry to characterise the charge storage ability of supercapacitors. In detail, we present a method to manufacture high surface area supercapacitor electrodes due to self-assembly of graphene flakes. To assemble a (flexible) supercapacitor, a filter paper, soaked in electrolyte, is sandwiched between two electrodes. As electrolyte, we use sulfuric acid and a mixture of sulfuric acid and vanadyl sulfate to prepare an EDLC and a hybrid supercapacitor, respectively. Characteristics of the voltammograms and the Nyquist plots due to the different charge storage mechanisms are discussed.

ELECTROPHILIC NON-COVALENT INTERACTIONS STUDIED BY SOLID-STATE NMR SPECTROSCOPY. **David L. Bryce**, University of Ottawa, Department of Chemistry and Biomolecular Sciences, Ottawa, ON K1N 6N5. (David.Bryce@uottawa.ca)

Research over the past decade has highlighted the widespread ability of elements across the periodic table to engage in electrophilic non-covalent interactions via electron donation to an area of elevated electrostatic potential and depleted electron density known as the sigma-hole. Prominent examples of these classes of interactions include halogen bonds, chalcogen bonds, pnictogen bonds, and tetrel bonds, where in each case the bond is named according to the identity of the atom accepting electrons. In this talk, I will highlight recent advances from our group on characterizing and understanding these various non-covalent bonds with multinuclear solid-state magnetic resonance as the main tool. Isotopes of interest from the NMR and NQR perspectives include e.g., ¹³C, ³¹P, ^{35/37}Cl, ⁷⁷Se, ^{79/81}Br, ¹¹⁹Sn, ¹²¹Sb, ¹²⁵Te, ¹²⁷I, etc.

I020 P

PETROLEUM COKE SOURCED ACTIVATED CARBONS FOR THE ADSORPTION OF ARSENIC (V) IN ACIDIC WATERS. **Kyle Fisher** and Andrew Vreugdenhil, Trent University, Department of Chemistry, 1600 West Bank Drive, Peterborough, ON K9L 0G2, Canada. (kylefisher@trentu.ca)

The removal of arsenic (V) from acidic waters was investigated using petroleum coke sourced activated carbons. Unmodified petroleum coke activated carbon, FeCl₃-loaded activated carbon (Fe-AC), KMnO₄-loaded activated carbon (Mn-AC), and a mixed FeCl₃-KMnO₄-loaded activated carbon (Fe-AC) was used for this evaluation. Surface characteristics of the activated carbons before and after arsenic adsorption were analyzed by x-ray photoelectron spectroscopy (XPS). Arsenic adsorption was significantly improved by the addition of a metal onto the surface of the activated carbon; increasing from 8.12% adsorption in the unmodified activated carbon to 50.93% in the FeMn-AC. Three adsorption mechanisms are proposed based on the XPS peak splitting of arsenic 2p3/2, iron 2p3/2, and manganese 2p3/2 observed. The first adsorption mechanism focuses on the change observed in Fe₂O₃ species on the surface of the activated carbon to FeOOH caused by the adsorption of arsenic. The second focuses on MnO to MnOOH species cause by arsenic adsorption. And the last focuses on MnO to MnO₂ species caused by arsenic adsorption. The three proposed adsorption mechanisms show how arsenic adsorption is increased due to the metal-loaded surface.

I021 P

PURSUIT OF J-COUPLINGS ACROSS CHALCOGEN BONDS IN DONOR-ACCEPTOR COMPLEXES WITH 1,2,5-CHALCOGENDIAZOLES AND PSEUDOHALIDES. Carina Almario and David L. Bryce, University of Ottawa, Department of Chemistry and Biomolecular Sciences, Ottawa, ON K1N 6N5, Canada. (calma032@uottawa.ca)

The chalcogen bond (ChB) refers to a non-covalent attractive interaction between an electrophilic chalcogen atom (group 16 elements) and at least one nucleophilic region in another molecular entity. The purpose of this study is to attempt to measure $J(^{125}\text{Te}, ^{15}\text{N}/^{13}\text{C})$ couplings in four ChB systems comprised of 3,4-dicyano-1,2,5-telluradiazole with various pseudohalide ChB acceptors using solid-state NMR (SSNMR) spectroscopy. Thus far, two cocrystals were fully characterized using powder X-ray diffraction, solution NMR, and ¹²⁵Te and ¹³C cross-polarization/high-power decoupling magic-angle spinning SSNMR to determine chemical shift tensors. The geometry of one cocrystal was characterized using single-crystal X-ray diffraction and the interatomic distance between KOCN and the ChB acceptor and Te donor atom through Te···O-C-N- and Te···N-C-O-systems were determined to be 3.001 Å and 2.512 Å, respectively. Furthermore, $J(^{125}\text{Te}, ^{15}\text{N})$ values are computed using density functional theory (DFT). Future work including the preparation of cocrystals with isotopically enriched cyanide to experimentally establish a correlation between ChB length and *J*-coupling data will be described.

I022

SOLID-STATE NMR SPECTROSCOPY FOR STRUCTURE CHARACTERIZATION AND ION-DYNAMICS STUDIES IN LITHIUM-ION-BASED SOLID-STATE ELECTROLYTES. **Sandamini H. Alahakoon¹**, Feipeng Zhao², Xiaona Li², Shumin Zhang², Jiamin Fu², Xueling Sun², Yining Huang^{1,*} 1. Department of Chemistry, University of Western Ontario, London, Ontario, N6A 5B7, Canada. 2. Department of Mechanical and Materials Engineering, University of Western Ontario, London, Ontario, N6A 5B9, Canada. (sulpanhe@uwo.ca)

The development of solid-state electrolytes (SSE) for all-solid-state Li-ion batteries (ASSLIB) provides a leap forward for safety and higher energy capacity compared to the conventional liquidelectrolyte-based Li-ion batteries, while globally reducing dependence on the fossil fuels to combat climate change. But, comparatively lower ionic conductivity in SSE to liquid-based electrolytes restricts the use of ASSLIB. Therefore, proper structure characterization and ion-dynamics studies are vital in the development of novel SSE. Solid-state NMR can help in understanding the relationship between local structure and ion-dynamic parameters including information on activation energies corresponding to short- and long-range Li-ion diffusions and jump rates.^[1] The unique information collected using novel high-performance SSEs will be presented in this study. In detail, the structural information collected using ¹¹⁹Sn, ³¹P, ¹¹⁵In, ³⁵Cl, ⁶Li, and ⁷Li magicangle spinning NMR experiments, Li-ion mobility studies including ⁷Li spin–lattice relaxation rates to determine the activation energies for Li-ion transport in different time-scales, and ⁷Li motional narrowing studies as a function of temperature will be discussed.^[2]

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I023 P

ENGINEERING OF MULTICOMPONENT COCRYSTALS AND ASSESSMENT BY SOLID-STATE NMR SPECTROSCOPY. **Mahée Côté** and David L. Bryce, University of Ottawa, Department of Chemistry and Biomolecular Sciences, Ottawa, ON K1N 6N5, Canada. (mcote118@uottawa.ca)

The engineering of multicomponent cocrystals is a fascinating and underdeveloped area of research. This research project aims to design and produce novel binary, ternary and quaternary cocrystals (multicomponent cocrystals) by harnessing the utility of a range of element-based non-covalent interactions including halogen bonds, chalcogen bonds, and hydrogen bonds, for example. Samples are prepared using green mechanochemical methods or slow solvent evaporation and the products are characterized by multinuclear solid-state magnetic resonance spectroscopy and powder X-ray diffraction. Mechanochemical methods include simple grinding with a mortar and pestle and ball milling. Single crystals are also grown when possible and single-crystal X-ray diffraction structures are determined. For example, we report here a novel polymorph of a 3:1 cocrystal of acridine and 1,3,5-trifluoro-2,4,6-triiodobenzene which features three crystallographically distinct nitrogen-iodine halogen bonds with distances ranging from 2.968 to 3.011 Å.

I024

EFFECT OF NITROGEN FERTILIZER APPLICATION ON PLANT USABLE NITROGEN SPECIES IN SOIL DURING SORGHUM GROWING SEASON ON MARGINAL LANDS IN ONTARIO. **Zhongwei Shi**; Hongbin Dong; Julia Lu, Toronto Metropolitan University, Department of Chemistry and Biology, 350 Victoria Street, Toronto, ON M5B 2K3, Canada. (julialu@ryerson.ca)

Fertilizer application is one of the most common agricultural practices that help plant growth and crop production. However, plants take up less than half of the fertilizer applied. The loss of fertilizer is increasing the cost of crop production and causing environmental pollution. This research focuses on water-soluble (i.e., plant usable) nitrogen species in Ontario marginal land soils during 2021 sorghum growing season and it contributes to a collaborative project "Develop a Low-Cost and High-Production System for Sorghum Biomass on Marginal Lands". Soil samples were collected from three study sites (London, Simcoe, Ottawa) at three different times (Before Planting, After Fertilizer Application, At Harvest) during 2021 sorghum growing season. The factors evaluated were land type (agricultural vs. marginal), fertilizer application, collection time, and sorghum hybrid. Water-soluble nitrogen species (NO3-, NO2-, and NH4+) were extracted following US EPA method 1312 and determined using colorimetry methods. The results indicate that nitrite and ammonium are below the detection limits in most of the samples. The concentration of nitrate, the predominant species in the soil, varies with the land type and collection time. Data analysis shows that heavy precipitation leached most of the nitrogen in marginal land soil, suggesting that nitrogen fertilizer serves as a source of water pollution.

CLEAN SYNTHESIS OF AMIDES BY SELECTIVE HYDRATION OF NITRILES IN WATER CATALYZED BY RECYCLABLE HETEROGENEOUS CATALYST BASED ON MOROCCAN OIL SHALE. **E. Ennesyry**, B. Mounir, M. Elkouali, M. Hamza, F. Bazi. Laboratoire de chimie analytique et moléculaire, Faculty of science Ben M'sick, Hassan II University of Casablanca, Boulevard Cdt Driss Harti, BP.7955, Ben M'sik, Casablanca, Morocco. (m.ennesyry@gmail.com)

Selective catalytic hydration of nitriles is considered the most efficient way to achieve the corresponding amides. It is an important transformation using the current technology for sustainable chemistry, which is usually catalyzed by metal complex catalysts. On the other hand, Moroccan oil shale was used in environmental applications by using it to develop adsorbent materials based on its mineral part to remove industrial colorants, heavy metals and radioactive elements and recently as heterogeneous catalyst in Knoevenagel condensation. To broaden the scale of applications of this raw material, we have developed a mesoporous material based on mineral part of oil shale by simple thermal treatment. Its analysis was carried out by different methods (XRD, MEB-EDS, BET-BJH, FX, FTIR) and its applicability as a heterogeneous recyclable and stable catalyst is demonstrated, which satisfies the criteria of environmental catalysts. This catalyst was used efficiently in selective catalytic hydration of nitriles to corresponding amides in water and the products were analyzed by ¹H ¹³C NMR for confirmation. The results clearly show that the catalyst developed can considered an efficient support for this transformation because of its capability to be active in water and recycled for several times.

I026

DEVELOPING PORTABLE ANALYTICAL METHODS USING FUNCTIONALIZED SURFACES AND ELECTROCHEMISTRY, **Zhe She**, Department of Chemistry, Queen's University, Kingston, ON, Canada. (<u>zhe.she@queensu.ca</u>)

Point-of-care systems have attracted significant research interest in recent years as they are designed to be portable and lower cost for a wide range of applications in detecting biological and chemical contaminations. These systems are important for many industrial sectors in environmental monitoring and clinical diagnostics. There are many promising methods under development using different types of materials and transducers. My group has been working on developing portable analytical methods using functionalized electrode surfaces. While self-assembled monolayers on gold surfaces are great platforms for creating different detection capability towards biological analytes such as bacterial whole-cells, toxin molecules and proteins, carbon-based electrodes have been tested for metal ion detections. [1-3] We are also interested in making small integrated devices using nanofabrication techniques and 3-D printers. I will share some of our development in these areas during this presentation.

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QUANTITATIVE ELECTROCHEMISTRY OF SUPRAMOLECULAR HOST-GUEST BINDING ON SURFACES. **Hua-Zhong Yu**, Simon Fraser University, Department of Chemistry, 8888 University Drive, Burnaby, BC V5A 1S6, Canada. (hogan_yu@sfu.ca)

Since 1980s the macrocyclic cucurbit[n]uril (CB[n]) hosts have shown dramatically increased research interests; their excellent guest recognition behavior leads to their great application potentials. For example, the inclusion complexes formed between CB[7] and various ferrocene (Fc) derivatives have extremely high binding affinities (10^9 to 10^{12} M⁻¹), which have been employed as an alternative of natural binding pairs (e.g., antigen-antibody, biotin-avidin) for fabricating versatile biosensing interfaces.

Based on both conventional cyclic voltammetry and advanced structural characterization, the binding thermodynamics and kinetics have been investigated on mixed ferrocenylundecanethiolate / octanethiolate self-assembled monolayers (SAMs) on gold. The results show that the inclusion binding behavior of this host-guest pair, while significantly affected by the surface, still has satisfactory stability for practical applications. The broad potential of this new interfacial Fc@CB[7] host-guest binding motif is manifested as nanoscale probes for the distribution of Fc terminal groups on SAMs, as an environmental regulator of long-range electron transfer process, and as an electrochemical sensor for pharmaceutical drugs via competitive host-guest assay strategy. It is expected that this new interfacial host-guest binding system can be further explored for fabricating well-controlled, ratiometric electrochemical biosensors.

I028

SPECTROFLUIDICS: A GENERALIZABLE FABRICATION METHOD OPENS THE DOOR TO A WIDE RANGE OF APPLICATIONS. **Jesse Greener**. Département chimie, Université Laval, Pavillon Alexandre-Vachon 1045, avenue de la médecine, local 4064^E, Québec, QC G1V 0A6, Canada. (jesse.greener@chm.ulaval.ca)

In this presentation, I will present the new class of "spectrofluidic" devices that are designed to enhance a range of on-chip studies of interest to our group. The approach merges attenuated total reflection (ATR) Fourier transform infrared (FTIR) spectroscopy with virtually any microfluidic design. A major design feature is the integration of the ATR element into the device rather than previous approaches where the ATR surface served as structural support for the entire device. The refocused role of the ATR as a dedicated analytical element results in excellent analytical performance. Spatially resolved measurements demonstrate the complementarity between design flexibility and high sensitivity. Multiple device designs have been prepared with this approach including: (i) limits of detection of glucose solutions rivaling those in the literature, (ii) applications to dynamic systems conditions, (iii) parallel measurements in multiple assay channels, (iv) characterization of complex multi-laminar co-flow streams, (v) on-chip monitoring of chemical reactions, (vi) monitoring carbon capture processes, and (vii) whole cell processes with catalytic bacteria. In addition to explaining the analytical chemistry and instrument development at the heart of the technology, I will explain a few of these exciting avenues that are now open thanks to this new technology. MASS SPECTROMETRY ANALYSIS OF BREAST CANCER-DERIVED EXOSOMES REVEALS DISEASE-SPECIFIC PHOSPHORYLATED ENZYMES. **Maxim V. Berezovski**, Zoran Minic, Nico Hüttmann, Suttinee Poolsup, Yingxi Li, Vanessa Susevski, Emil Zaripov Department of Chemistry and Biomolecular Sciences, University of Ottawa, Ottawa, ON K1N 6N5, Canada. (maxim.berezovski@uottawa.ca)

We performed a phosphoproteomic analysis of breast cancer-derived exosomes to provide insight into the molecular and cellular regulatory mechanisms important for breast cancer tumor progression and metastasis [1]. We examined three cell line models for breast cancer: MCF10A (non-malignant), MCF7 (estrogen and progesterone receptor-positive, metastatic), and MDA-MB-231 (triple-negative, highly metastatic). To obtain a comprehensive overview of the exosome phosphoproteome derived from each cell line, effective phosphopeptide enrichment techniques IMAC and TiO2, followed by LC-MS/MS, were performed. Among 855 distinct phosphoproteins, we validated four enzymes associated with cancer and present only in exosomes isolated from MCF7 and MDA-MB-231 cell lines: ATP citrate lyase (ACLY), phosphofructokinase-M (PFKM), sirtuin-1 (SIRT1), and sirtuin-6 (SIRT6). With the exception of PFKM, the specific activity of these enzymes was significantly higher in MDA-MB-231 when compared with MCF10A-derived exosomes. This study demonstrates that exosomes contain functional metabolic enzymes that could be further explored for their potential use in early breast cancer diagnostic and therapeutic applications.

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I030

APPLICATIONS OF MASS SPECTROMETRY FOR THE STUDY OF THE HUMAN GUT MICROBIOME. **Daniel Figeys**, School of Pharmaceutical Sciences, Faculty of Medicine, University of Ottawa. (<u>dfigeys@uottawa.ca</u>)

The gut microbiome is important to our health and is implicated in a growing list of diseases. The gut microbiome interacts with the host through the production of enzymes, peptides and metabolites. Our understanding of the function of the microbiome is limited as the vast majority of microbiome studies have focused on genomic approaches. Fortunately, mass spectrometry can be used to study the function of the microbiome and, in particular, the metaproteome, its regulations through post-translational modifications and metabolites produced by the microbiome. Here we will discuss recent applications of metaproteomics combined with metabolomics to study the microbiome in pediatric inflammatory bowel disease and microbiomedrug interactions.

ESI-MS ANALYSIS OF GUEST-HOST COMPLEXES TOWARDS INHIBITING CRYSTALLIN AGGREGATION AND CATARACTS. **Dev Seneviratne**¹, Dr. Ngong Kodiah Beyeh², Dr. Sanela Martic^{1*}. ¹Department of Forensic Science, Environmental and Life Science program, Trent University, Canada. ²Department of Chemistry, Oakland University, Rochester, MI, USA. (*sanelamartic@trentu.ca)

People over 60 years old suffer from cataracts due to many causes. The key reason is the crystallin protein-derived peptide α A- 66-80 aggregation. While cataract is operable, in certain countries surgery may not be accessible or financially feasible. In addition, there is a waiting period prior to surgery which is debilitating. To overcome current challenges related to cataracts, anti-cataract drugs would be ideal. Herein, we report on the use of the Host-Guest chemistry as a great platform for drug design. We developed the synthetic host molecules that bind to pathogenic α A- 66-80 peptide and inhibit aggregation (i.e. cataracts formation). ESI-MS was used to characterize the host, peptide, and host-guest complexes. The complexes were formed between negatively charged hosts and a peptide. All host-peptide complexes were effective inhibitors of aggregation. Understanding the interactions between the host molecules with pathogenic peptides and proteins may inform the development of improved therapeutic treatment for cataracts and other diseases, which remain without a cure.

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I032

MALDI MASS SPECTROMETRY IMAGING – CHALLENGES AND OPPORTUNITIES IN QUANTITIVE ANALYSIS. **Ken Yeung**. Western University, Departments of Chemistry and Biochemistry, London, ON N6A 5B7, Canada. (kyeung@uwo.ca)

Tissue imaging by mass spectrometry (MS) is performed by recording the masses of all desorbed ions coordinated to the two-dimensional space of a tissue section. From the collective data, one could extract the spatial distribution of an ion of interest by displaying its intensity in 2-dimensions in the form of a heat map. MS imaging (MSI) is a very promising tool in biochemical research, for being a universal and label-free imaging technique. When interpreting these heat maps, one expects MS signal intensity to only correlate with analyte concentration present in the sample, but this is not always the case. In MSI based on matrix-assisted laser desorption/ionization (MALDI), the matrix plays a critical role in transfer the energy from the laser pulsed beam to the analyte for desorption/ionization. A carefully optimized matrix can selectively facilitate ionization of the analyte of interest. However, the matrix can also have unintended influences of the signal intensities, such as the unwanted fragmentation of analytes or introduction of additional signal variability related to application procedures of the matrix on biological tissues. In this presentation, the non-quantitative nature of MSI based on MALDI and its subsequent limitations will be presented along with progress in addressing the issues.

I033

ABSOLUTE ELECTROCHEMILUMINESCENCE QUANTUM EFFICIENCY. Kenneth Chu, Jonathan Adsetts, Liuqing Yang, **Zhifeng Ding.** Department of Chemistry, The University of Western Ontario, London, ON N6A 5B7, Canada (zfding@uwo.ca)

Electrochemiluminescence (ECL) is the process in which electrogenerated radicals form excited species that emit light without the need for an external light source. ECL is a powerful analytical technique that is fast, highly sensitive and selective, requires low quantity and is cost effective. ECL quantum efficiencies (Φ_{ECL}) have been determined relative to Ru(bpy)₃²⁺ under similar conditions, regardless of whether the conditions are favorable for Ru(bpy)₃²⁺ emissions or not. We present here our strategies to use a common disk electrode, spectrometer, and photomultiplier tube to measure the Φ_{ECL} . The more common light detection hardware and electrodes combined with an in-depth calculation walkthrough will provide ECL researchers the necessary tools to implement Φ_{ECL} measurement procedures in their own laboratories.

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I034

TRANSFER AND PERSISTENCE ANALYSIS OF GUNSHOT RESIDUE BY MP-AES AND ICP-MS. **Deanna Haas**¹, Kiana Martin¹, Matthew De Vries¹, Shannon Accettone^{1,2}, Sanela Martic^{1*}, David Ruddell³. ¹Department of Forensic Science, Environmental and Life Sciences, ²Department of Chemistry, Trent University, 1600 W Bank Drive, Peterborough, ON K9L 0G2, Canada, ³Centre of Forensic Sciences, 25 Morton Shulman Ave, Toronto, ON M3M 0B1. (deannahaas@trentu.ca) (*sanelamartic@trentu.ca)

Gunshot residue (GSR) involves the deposition of metal particles emitted from a firearm.1 The combination of lead, barium, and antimony is characteristic of GSR particles.1 Among the challenges in using this evidence is the transfer and persistence of these particles. Specifically, the transfer and persistence of GSR on fabrics are of forensic relevance (primary, secondary or tertiary transfers). The detection and quantification at these trace metal levels are required for evidentiary analysis of gun-related crimes.1,2 Herein, metal transfer and persistence on fabrics were evaluated using MP-AES and ICP-MS instruments. Cotton fabrics were contaminated using mixed metal solutions, and particle transfer was simulated using contact, pressure, time, and agitation to mimic GSR transfer. The extraction protocols were optimized and samples were analyzed using both instrumental methods. The transfer was high with applied pressure and less so with contact. All metals were found during transfer studies. GSR soiled samples were also tested by similar means. Data presented will provide additional insight into GSR transfer and persistence.

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I035

GENERATING ELECTRODES USING 3-D PRINTING TECHNOLOGY FOR BIO-ANALYTICAL APPLICATIONS. **Kristin Partanen**, Phillip Hillen, Kevin McEleney, Richard Oleschuk*, Zhe She*. Queen's University, Department of Chemistry, Kingston, ON K7L 3N6, Canada. (Kristin.partanen@queensu.ca)

Bacterial and viral detections are important in environmental monitoring and clinical diagnostics. Current methods, such as culture tests and polymerase chain reactions, are successful when detecting bacteria and viruses, but are costly, require trained personnel, take 2 to 5 days to receive results, and are inaccessible in resource poor settings.1 Therefore, it is important to move towards point-of-care testing (POCT) to decrease the detection time of bacterial species. There is a growing interest towards electrochemical (EC) sensors, such as the glucose meter, as they are based on compact potentiostats which offer portable and low-cost solution for POCT.2 For an effective EC sensor, an electrode chip needs to be fabricated for sensing. Techniques, such as electron beam lithography and photolithography, have been used to design features on chips but they are costly, time-consuming, and may not be accessible. 3 Therefore, there is a need to investigate new methods of fabricating chips that are capable for sensing applications. By using 3-Dimensional (3-D) printing technology, this can reduce the costs of chip fabrication and allow for tailored designs easily.

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I036

DIGITAL MICROFLUIDICS COMBINED WITH MASS SPECTROMETRY FOR SINGLE-CELL PROTEOME ANALYSIS. **Jiaxi Peng**, Erica Y. Scott, Calvin Chan, Maxwell E. Olson, Aaron R. Wheeler. Department of Chemistry, University of Toronto, Toronto, ON Canada. (jiaxi.peng@mail.utoronto.ca)

Proteomics, at the single cell level, allows global observation of intracellular protein composition and changes. However, highly sensitive and spatial-resolved proteome analysis of single cells are restricted by conventional sample processing methods due to challenges associated with handling single cells *in-situ*. A cutting-edge microfluidic platform called DISCO (Digital microfluidic Isolation of Single Cells for -Omics) was developed in this work. Combined with mass spectrometry (MS), this DISCO-based MS platform (DISCO-MS) enables automated single cell sorting, lysis, collection, and identification. Based on this DISCO-MS platform, the target single cells of interest can be selected for lysis, and then their heterogenous cellular proteome can be identified to elucidate the cell states. We assessed the number and relative intensity of proteins identified in single neural stem cells and matched the proteome to their cell phenotype, respectively. Proteomic differences of neural stem cells were studied, which demonstrated the capacity of this DISCO-MS platform to identify proteins involved in a wide range of cellular pathways and further stretching the utility for the identification of intact proteomes from single cells. By using this DISCO-MS platform, investigating proteome at single-cell level may facilitate the accurate analysis of cellular heterogeneity for better diagnostics and therapy. APPLICATION OF ANALYTICAL TOOLS TO PROBE THE BIONORGANIC CHEMISTRY OF TOXIC METAL(LOID)S IN MAMMALS. M. Doroudian, A. Gautam, T.G. Bridle and J. Gailer, University of Calgary, 2500 University Drive NW, Calgary, AB T2N 1N4, Canada. (jgailer@ucalgary.ca)

All organisms must be supplied with sufficient amounts of essential elements via their diet/ drinking water for the *in vivo* assembly of biochemically vital metalloproteins. Human activities, however, increasingly contaminate the diet/drinking water with potentially toxic metal(loid)s which can adversely affect the assembly of metalloproteins. Thus, the development of analytical methods to rapidly quantify metalloproteins in biological fluids represents a feasible approach to gain insight into the dyshomeostasis of essential elements in individual organisms. To this end, a brief overview will be presented about analytical methods we have developed to quantify metalloproteins in blood plasma [1, 2]. In addition, select examples will be presented about how these methods have been applied to gain important new insight into the toxicological chemistry of environmentally ubiquitous mercury species that unfold in blood plasma [3] and red blood cell cytosol [4].

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I038

SEPARATION ORTHOGONALITY IN 2D HPLC – CZE APPLICATIONS FOR BOTTOM-UP PROTEOMICS. Darien Yeung, Vic Spicer, **Oleg Krokhin.** University of Manitoba, 799 JBRC, 715 McDermot ave., Winnipeg, MB R3E 3P4, Canada. (oleg.krokhine@umanitoba.ca)

Evaluation of peptide separation orthogonality in 2D LC-LC applications has been performed in recent years using ever growing datasets of tryptic peptides [1]. Based on availability of these data for the identical tryptic digest of yeast and superior accuracy of SSRCalc CZE prediction model [2] this work aimed at determining separation orthogonality for 20 various LC-CZE applications. Separation orthogonality has been accessed by both R² and filling % methods using 10,000 peptide collections of retention data for 20 different peptide HPLC separation modes. CZE retention time values were calculated using SSRCalc CZE model [2]. We found that R² value correlations vary from 0.0007 (RPLC pH 10) to 0.74 (worst for SCX). Filling percentage values varied between 77-79% (best for RPLC pH 10, RPLC HFBA) and 45% (worst for SCX). These findings are not surprizing given that both CZE and SCX are peptide's charge and size driven separation methods. Considering overall robustness of RPLC separation mode, we recommend those for 2D LC-CZE combinations. RPLC with HFBA as ion pairing additive represents the most favorable option due to higher peptide retentivity.

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NUCLEAR MAGNETIC RESONANCE STUDY OF A NOVEL TETREL BONDED COCRYSTAL. Sachin Nivantha Liyanage and David L. Bryce, University of Ottawa, Department of Chemistry and Biomolecular Sciences, Ottawa, ON K1N 6N5. (sliya071@uottawa.ca)

The tetrel bond (TtB) is a directional noncovalent interaction between an area of elevated electrostatic potential on a Group 14 atom (i.e., C, Si, Ge, Sn, Pb, Fl) and an electron donor, such as the lone pair of a Lewis base or an anion. The purpose of this work is to measure the J(¹¹⁹Sn,¹³C/¹⁵N) coupling across a tetrel bond in a series of novel cocrystals. Slow evaporation was used to cocrystallize triphenyltin chloride (TtB donor), potassium cyanide (TtB acceptor), and cryptand-222 to obtain potential tetrel bonded cocrystals. A simple solution cocrystallization approach was also conducted with triphenyltin chloride, potassium cyanide, and 18-crown-6, yielding various possible cocrystals. These samples were analyzed through ¹³C, ¹⁵N, and ¹¹⁹Sn cross-polarization magic-angle spinning solid-state NMR spectroscopy, powder X-ray diffraction, and single-crystal X-ray diffraction to verify the formation of tetrel bonds to tin. Progress to date in analyzing the ¹³C, ¹⁵N, and ¹¹⁹Sn solid-state NMR spectra will be described.

I040

SELECTIVE DETECTION OF SARS-COV-2 USING LOCALIZED SURFACE PLASMON RESONANCE APTASENSOR. **Amanda Oake**,¹ Tyra Lewis,² Erin Giroux,² Sanela Martic*² ¹Fleming College, Peterborough, ON, ²Department of Forensic Science, Environmental and Life Sciences, Trent University, Peterborough, ON. (amanda.oake@flemingcollege.ca) (*sanelamartic@trentu.ca)

Increased demands in clinical testing during the current COVID-19 pandemic has demonstrated the immediate need for valuable and efficient solutions for detection of the severe acute respiratory beta-coronavirus 2 (SARS-CoV-2). Conventional testing methods include polymerase chain reaction (PCR) or serological antibody detection, but these methods can be costly, labor-intensive and time-consuming. Thus, alternative testing methods are needed that would also improve the overall reliability, accuracy, and response time for the acquisition of results. Viral antigens such as SARS-CoV-2 spike (S) glycoprotein play a vital role in the functioning of the virus and can be used as a diagnostic biomarker for determination of viral infection. For biosensing applications, aptamers are suitable high-affinity and cost-effective binding partners for specific targets, such as viral antigens. Localized surface plasmon resonance (LSPR), offers real-time, rapid results within minutes which is beneficial for point-of-care testing. Herein, a 2-channel LSPR aptasensor system was fabricated using ssDNA aptamers for the detection of SARS-CoV-2 antigens.¹ The S1 aptamer was found to selectively bind to the SARS-CoV-2 S1 antigen with high affinity. Kinetic parameters and binding affinity (ka, kd, KD) were determined for the aptamer-antigen assay. Spiked saliva samples achieved >90% recovery when tested with the S1 aptasensor, and the sensor exhibited excellent shelf-life stability. Data indicate that LSPR is a viable tool for measuring SARS-CoV-2 related aptamer-antigen interactions and this strategy may be applied to other viral or non-viral antigen targets.

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IR NANOSPECTROSCOPIC INVESTIGATION ON SOLVATION LAYERS ON NANOSTRUCTURED INTERFACES OF SELF-ASSEMBLED MONOLAYERS. **Nafiseh Samiseresht**, Petra Ebbinghaus, Anne Jakubek, Martin Rabe, Department of Interface Chemistry and Surface Engineering, Max-Planck-Institut für Eisenforschung GmbH (MPIE), Max-Planck-Str.1, 40237 Düsseldorf, Germany. (n.samiseresht@mpie.de, m.rabe@mpie.de).

The structure of interface water/solvation layers is thought to affect surface reactions such as bioadhesion [1]. In this study oligo (ethylene glycol) self-assembled-monolayers (OEG terminated SAMs) with hydrophilic character on the surface of gold (111) is employed to be investigated under elevated relative humidity by nano IR spectroscopy. Knowing that OH-stretching modes reveal information about the strength of interface hydrogen bonds, a particular vibrational spectroscopic technique 'atomic force microscopy infrared spectroscopy (AFMIR)' is employed to provide spectral and chemical information in the ranges of <1900 cm⁻¹ and 3900- 2700 cm⁻¹ using QCL and OPO lasers [2]. OEG islands of different molecular conformation were achieved by varying the solution concentration and immersion time during SAM formation. Detailed analysis of the CH₂ and CH₃ stretching modes around 3000 cm⁻¹ reveal structural details in the nano domain about the conformation of OEG moieties and the OH-stretching bands of adsorbed water on the SAMs reveal distinct information about the hydrogen bonding state of the adsorbed molecules and hence the structure of interfacial water solvent.

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I042 P

DO ENANTIOMERS GIVE IDENTICAL SOLID-STATE NMR SPECTRA? Audrey-Anne Lafrance, Manon Girard, and David L. Bryce, University of Ottawa, Department of Chemistry and Biomolecular Sciences, Ottawa, ON K1N 6N5. (alafr043@uottawa.ca)

In the vast majority of practical situations, enantiomers should provide identical NMR spectra. Theoretically predicted differences in J coupling constants and chemical shifts for different enantiomers are typically orders of magnitude smaller than what is measurable experimentally. Prompted by recent controversy in the literature concerning purportedly different NMR signal intensities arising from L- and D-amino acid powders under cross-polarization magic-angle spinning (CP/MAS) conditions, we report here a systematic study of the factors affecting reproducibility in simple CP/MAS experiments. In addition to instrumental settings (e.g., contact time), such factors include crystallite size, polymorphism, T1 values, and how the sample is packed in the rotor. Challenges faced in ensuring exact and consistent reproducibility of NMR signal intensity even for the same enantiomer place limitations on the level of precision in peak areas one may reasonably expect for different enantiomers. Experimental ¹³C and ¹⁵N NMR data are presented for selected L- and D-amino acids and possible reasons for minor differences in signal intensity are explored and discussed.

WHAT THE ELK?! BIOMONITORING OF ENVIRONMENTAL SAMPLES IN BUTTE, MONTANA TO DETERMINE THE IMPACT FROM A LOCAL SUPERFUND SITE. **Nausheen Sadiq**¹, Catharina Venter¹, Kevin Hayes², James Walker², Caleb Marx² and Gwen O'Sullivan². ¹ Mount Royal University, Department of Chemistry and Physics, 4825 Mount Royal Gate SW, Calgary, T3E 6K6, Canada (nsadiq@mtroyal.ca). ² Mount Royal University, Department of Earth and Environmental Science, 4825 Mount Royal Gate SW, Calgary, T3E 6K6, Canada. (nsadiq@mtroyal.ca)

One of the largest superfund sites in the United States is located in Butte, Montana [1]. Previously a copper mine, the Berkeley Pit is now a part of the Silver Bow Creek/Butte Area Environmental Protection Agency Superfund site [1]. Since there are deep-rooted problems regarding toxic levels of metals in the area, we will study the impact on the local food web. Past research has focused on contaminants in water [1], encouraging this study to conduct biomonitoring in plant material, soil, water, and elk (antlers, hide, and scat) samples. Elk byproducts will be used as an indicator as they are expected to contain high levels of heavy metals due to elk eating habits and the tendency of metals to readily move through water, soil, and plant material. An Agilent 7850 inductively coupled plasma-mass spectrometer (ICP-MS) was utilized in this study to analyze arsenic, lead, iron, cadmium, aluminum, copper, zinc, mercury, selenium, nickel, chromium, manganese, vanadium, cobalt, silver and tin. This study will determine the burden this Superfund site has on the elk population and the subsequent risk to human health and safety due to elk meat consumption.

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I044

SELENIUM-77 AND TELLURIUM-125 SOLID-STATE NMR AND X-RAY DIFFRACTION STUDY OF CHALCOGEN-BONDED 3,4-DICYANO-1,2,5-CHALCOGENODIAZOLE COCRYSTALS AND POLYMORPHS. **Tamali Nag**, Jeffrey S. Ovens, and David L. Bryce, University of Ottawa, Department of Chemistry and Biomolecular Sciences, Ottawa, ON K1N 6N5, Canada. (<u>tnag094@uottawa.ca</u>)

A chalcogen bond is an electrophilic interaction between an area of depleted electron density and elevated electrostatic potential (σ -hole) on a chalcogen atom, and an electron donor. Here, the preparation of a series of novel cocrystals featuring 3,4-dicyano-1,2,5-selenodiazole and of 3,4-dicyano-1,2,5-tellurodiazole as chalcogen bond donors and dimethyl sulfoxide & tetraphenylphosphonium bromide as chalcogen bond acceptors is reported. These novel cocrystals have been characterized by ⁷⁷Se/¹²⁵Te solid-state NMR spectroscopy, single-crystal X-ray diffraction, and powder X-ray diffraction. The two σ -holes of each chalcogen in the donor molecules enable directional chalcogen-bond-driven cocrystallization with a range of electron donors. The halide acceptor, when cocrystallized with the chalcogen bond donor, exhibits two polymorphic forms as confirmed by single crystal X-ray structures. ⁷⁷Se and ¹²⁵Te solid-state NMR spectroscopy reveals in some cases an interesting spectral pattern where for one apparently crystallographically unique Se/Te site, a multiplet is observed. The possible origins of this complex multiplet are explored.

STEREOSELECTIVE ANALYSIS OF TARTARIC ACID USING COMPLEXATION WITH EUROPIUM-TETRACYCLINE AND SEPARATION BY CAPILLARY ELECTROPHORESIS **Douglas B Craig**, Joshua W Hollett, Sumaiya Abas, Brynne K Riehl, Chemistry Department, University of Winnipeg, Winnipeg, Manitoba, Canada (d.craig@uwinnipeg.ca)

Complexes of D-, L- and DL-tartaric acid were made with Eu^{3+} -tetracycline and separated using capillary electrophoresis coupled to laser-induced fluorescence detection. The separation was performed in TRIS-HEPES buffer containing 40% formamide in a 25 cm long capillary and with a separation voltage of 20 kV. Retention time for the complexes was 66 s. A complex was detected with the 'un-natural' D- and DL-isomers but not with the 'natural' L-isomer. Complex with the D-isomer was detected at a concentration of 5 μ M. In the absence of a separation, all three stereoisomers formed complexes. Formation of the complex is dependent upon the presence of organic solvents. Further studies showed that during the separation period all of the complexes start to dissociate, and in the case of the L-isomer the complex is completely lost. Rate of loss is temperature dependent and the activation energies of dissociation were determined. Molecular modelling of the complexes has been undertaken. As an application, the concentration of D-tartaric acid in loquat fruit, one of the very few natural sources of D-tartaric acid, was determined.

I046 P

HALOGEN BONDS CATALYZE METHYL GROUP ROTATIONS. **Teodor Iftemie**, Shubha Gunaga, and David L. Bryce, University of Ottawa, Department of Chemistry and Biomolecular Sciences, Ottawa, ON K1N 6N5, Canada. (tifte016@uOttawa.ca)

Intramolecular and intermolecular bonding can affect the dynamic character of molecules [1]. For instance, we see a great reduction in the energy barrier of methyl group's rotation when a molecule has undergone hydrogen bonding [2]. Similar results are obtained with van der Waals interactions [3], and with halogen bonding, which acts as an even greater catalyst than hydrogen bonds [2]. In this report, we study how halogen bonds in cocrystals impact the methyl rotational barrier using 2H solid-state NMR relaxation experiments. By using multiple techniques of crystallization, we produced several novel cocrystals for our study: as halogen bond acceptors, we use 3,4,7,8-tetramethyl-1,10-phenanthroline, 4,4'-dimethoxyoctafluorobiphenyl, and 4-methylaminopyridine, with iodinated and brominated halogen bond donors. Density functional theory calculations are carried out to support the experimental results.

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METRICS AND MODELING FOR PORTABLE ANALYTICAL INSTRUMENTS. Suzanne Schreyer, Senior Scientist, Rigaku Analytical Devices. (Suzanne.Schreyer@rigaku.com)

Portable instrumentation is an increasingly useful tool for identification, verification and quality control in pharmaceutical, chemical and agricultural industries. However, the methodology behind developing a robust application for material identification or quantitative analysis is often poorly understood or poorly implemented. A properly designed chemometric approach to data analysis is required in order to develop models that can be consistently applied and will stand the test of time. Along with properly designed sampling approaches, an analytical approach to model development and the correct use of chemometric tools is essential to identification models, and especially for quantitative analyses. Examples will be provided to illustrate some of the approaches required to develop robust models for both identification and quantitative analysis on portable spectroscopic instruments. These include (1) qualitative pharmaceutical and chemical library identification methods, and (2) quantitative food and agricultural methods.

I048

RATIONAL DESIGN AND DEVELOPMENT OF SEROLOGICAL DIAGNOSTICS BY IMMUNOPRECIPITATION-TARGETED MASS SPECTROMETRY. Zhiqiang Fu, Yasmine Rais, Delaram Dara and **Andrei P. Drabovich**, Division of Analytical and Environmental Toxicology, Department of Laboratory Medicine and Pathology, Faculty of Medicine and Dentistry, University of Alberta, Edmonton, Alberta T6G 2G3, Canada. (drabovic@ualberta.ca)

Serological tests, such as indirect immunoassays for detection of anti-viral antibodies in blood serum, are currently designed to facilitate fast assay development and high throughput, but suffer from semi-quantitative measurements, lack of standardization and cross-reactivity. Here, we suggested that combination of immunoaffinity enrichments with targeted mass spectrometry measurements would enable rational design of serological diagnostics of infectious diseases, such as COVID-19, and resolve immunoassay limitations. A multiplex immunoprecipitation-selected reaction monitoring (IP-SRM) assay enabled differential quantification of anti-SARS-CoV-2 antibody isotypes (IgG, IgA, IgM, IgE, IgD) and subclasses (IgG1-4, IgA1-2). Evaluation of numerous antigen-antibody subclass combinations revealed receptor-binding domain (RBD)-IgG1 as a combination with the highest diagnostic performance. Further validation revealed that anti-RBD IgG1, IgG3, IgM and IgA1 levels were significantly elevated in COVID-19-positive plasma, while IgG2, IgG4, and IgA2 levels were not informative. Anti-RBD IgG1 provided 100% diagnostic specificity and sensitivity to detect COVID-19 convalescent plasma at 385 ng/mL cutoff [1]. Immunoprecipitation-targeted mass spectrometry assays will facilitate improvement and standardization of serological diagnostics and offer novel tools for the comprehensive evaluation of antibody subclass cooperation in immunity response.

[1] Fu, Z.; Rais, Y.; Drabovich, AP. Immunoprecipitation-targeted proteomics assays facilitate rational development of SARS-CoV-2 serological diagnostics. *medRxiv*, 2021, https://doi.org/10.1101/2021.10.25.21265408

I049

ARSENIC SPECIATION IN THE ENVIRONMENT. Qingqing Liu, Xiufen Lu, Tetiana Davydiuk, Xiaojian Chen, Karen S. Hoy, Chester Lau, Jordan R.M. Schofield, and **X. Chris Le** Division of Analytical and Environmental Toxicology, University of Alberta, Edmonton, Canada. (xc.le@ualberta.ca)

Determining arsenic speciation in the environment is important because the toxicity and environmental effects of arsenic vary greatly with its chemical speciation. Food and water are main sources of human exposure to arsenic. This presentation focuses on our recent work using liquid chromatography and mass spectrometry to identify and quantify arsenic species in food, including fish. Arsenic species were extracted using water/methanol and with enzyme treatment. Arsenic species in the extracts were separated using high performance liquid chromatography (HPLC) and detected using inductively coupled plasma mass spectrometry (ICPMS). Electrospray ionization tandem mass spectrometry (ESI MS/MS), used in combination with HPLC and ICPMS, provided complementary information for the identification and characterization of arsenic species. Analysis of krill oil, tuna fillets, and kelp revealed the presence of more than 20 arsenolipids,^[1] including novel arsenic-containing fatty acids, arsenic-containing hydrocarbons, and arsenic-containing phosphatidylcholines not previously reported. Identification of arsenic species were supported by HPLC peaks detected with both ICPMS and ESI MS. Method validation using available arsenic standards and successful identification of previously unknown arsenolipids in food samples demonstrated the potential for environmental research using HPLC separation and simultaneous detection with both ICPMS and ESI MS/MS.

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I050

SEPARATION AND MASS SPECTROMETRY TECHNIQUES IN WATER RESEARCH. Caley Craven, Nicholas Wawryk, Kristin Carroll, and **Xing-Fang Li**, Division of Analytical and Environmental Toxicology, University of Alberta, Edmonton, Canada. (xingfang@ualberta.ca)

Disinfection of drinking water effectively prevents waterborne disease. An unintended consequence of the chemical disinfection process is the formation of disinfection by-products (DBPs). Epidemiological studies have indicated potential chronic adverse health effects of DBPs. However, it remains unclear which DBPs cause the observed effects. Odor and off taste of drinking water are additional challenges for water industry. Majority of DBPs are un-identified to date. Therefore, we aim to develop innovative analytical approaches to address water quality issues. Our current research focuses on the development of advanced separation and mass spectrometry techniques for identification of reactive nitrogenous organics in source water and their DBPs in treated drinking water. Reactive organic nitrogen can produce both toxic and odorous DBPs. We will present an example of developing a SPE-HILIC-MS/MS method for tracing amino acids in source water. An application of this method to water analysis during the springs of 2021 and 2022 reveals a correlation between the occurrence of amino acids in source water and the formation of toxic DBPs as well as tap water odor complaints. We will discuss the implication and significance of these findings for water treatment.

ELECTROCHEMILUMINESCENCE AND SPECTROSCOPIC INSIGHTS INTO METALLIC NANOCLUSTERS. Kenneth Chu, Mahdi Hesari, Zhifeng Ding, Department of Chemistry, The University of Western Ontario, London, ON N6A 5B7, Canada (kchu59@uwo.ca)

Electrochemiluminescence (ECL) is the process in which electrogenerated radicals form excited species that emit light without the need for an external light source. ECL is a highly versatile electrochemical tool that can be used in combination with other spectroscopic techniques to yield mechanistic insights of light-emitting processes. We present our characterization of several novel metallic nanoclusters, where electrochemiluminescence and time-resolved spectroscopy were used to study excited state generation and subsequent light emission. In particular, we discovered a fundamental relationship between the structure of these nanoclusters and their electrochemiluminescence quantum efficiencies, which were among the highest observed for this class of nanomaterial.

I052

EFFECT OF STORAGE CONDITIONS ON ACTIVITY OF GLUTARALDEHYDE-CROSSLINKED TRYPSIN USING DESIGN OF EXPERIMENTS (DOE) AND CE-BASED PEPTIDE MAPPING WITH TWO-MARKER MIGRATION TIME CORRECTION. Marie-Pier Ouellet, Meriem Boudhina and **Karen C. Waldron.** Department of Chemistry University of Montreal, CP 6128, succ. Centre-ville, Montréal, QC, H3C 3J7, Canada. (karen.waldron@umontreal.ca)

Rapid enzymatic digestion for bottom-up proteomics can be achieved with insoluble proteolytic enzymes, which are reusable and compatible with microfluidics. Our group uses trypsin crosslinked with glutaraldehyde (GA) to make insoluble enzyme particles that don't require a solid support and can be adapted to *in-situ* microreactors. Our multi-step enzyme immobilization procedure developed in 2004 involves 15 washing steps. With a Design of Experiments (DoE) approach, this could be reduced to 9 steps without significant loss in activity as evaluated by capillary electrophoresis (CE)-based peptide mapping of lysozyme digests. In the current study, we applied DoE to investigate three parameters related to storage of a suspension of GA-trypsin particles at 4 °C: NaCl content (50-150 mM), phosphate buffer strength (0-140 mM) and duration (0-10 days). Activity of each stored enzyme was evaluated by digesting aliquots of the same batch of denatured lysozyme and comparing their peptide maps obtained by CE at pH 2.5. Since CE can suffer from poor migration time reproducibility, a two-marker correction method adapted from Li et al. [1] was used to improve reliability of using CE-based maps to indirectly assay GA-trypsin activity. Because peptides vary in number and intensity with GA-trypsin activity, external markers that could be monitored at a different wavelength than tryptic peptides were chosen: 1,10phenanthroline and nicotinic acid. An in-house Igor ProTM procedure was written for two-marker alignment of peptide maps. The user simply inputs the migration times of each marker in the first and second electropherograms being compared, the second trace gets aligned to the first and both peptide maps are re-plotted. The procedure was applied sequentially to the digests to compare the number and intensity of peptides, which infers GA-trypsin activity. These values were used in the DoE method to optimize storage conditions for GA-immobilized trypsin.

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MEASUREMENT ERROR MODELS FOR LC-MS PROTEOMICS MEASUREMENTS. Seketoulie Keretsu ^{1,3} and **Tobias K. Karakach^{1,2}**, ¹Laboratory of Integrative Multi-Omics Research, Department of Pharmacology, Dalhousie University, Halifax, NS, B3H 4R2, Canada, ²Beatrice Hunter Cancer Research Institute, Halifax, NS, B3H 4R2, Canada, ³Department of Pathology, Dalhousie University, Halifax, NS, B3H 4R2, Canada. (karakach@dal.ca)

Proteomics by LC-MS yields data that are complex and advanced computational tools are required to deconvolve this complexity. Alternatively, test statistics under a generalized least squares framework, are used to model the relationship between the instrumental response (y_i) and the biological question (x_p) under the null hypothesis that the coefficients, $\beta_j s$, are equal to zero *i.e.*, $y_i = \beta_j x_p + \epsilon_i$. Fundamentally, it is assumed that the data are normally distributed while the errors $(\epsilon_i s)$ are uniformly distributed. These assumptions are not always true, however, and several approached to transform the data to meet these assumptions have been devised.

We comprehensively characterize measurement uncertainties associated with LS-MS proteomics using an experiment designed to capture contributions of several sources. We show that for a well-designed experiment, the total variance associated with biological are hierarchical such that $\sigma_{total}^2 = \sigma_{biol}^2 + \sigma_{tech}^2$, where $\sigma_{biol}^2 \gg \sigma_{tech}^2$. We also show the multivariate structure of the noise depicting correlations between variables and devise a method to model this noise allowing errors in LC-MS measurements to be accurately estimated without the need for extensive replication. This estimate is then used to pre-process proteomics data to allow low intensity signals to be modelled with the same relative importance.

I054 P

PROBING THE FORMATION OF TOXICOLOGICALLY RELEVANT CD-CYSTEINE AND CD-HOMOCYSTEINE COMPLEXES BY HPLC-FAAS, Amanda Gomez, **Astha Gautam**, and Jürgen Gailer, Department of Chemistry, University of Calgary, 2500 University Drive NW, Calgary, AB T2N 1N4, Canada. (agautam@ucalgary.ca)

Chronic exposure of humans to Cd via the consumption of contaminated foods can adversely affect the kidneys and is associated with the etiology of type 2 diabetes [1]. The bioinorganic processes of Cd2+ that unfold in the bloodstream, however, are incompletely understood. Although Cd2+ binds to human serum albumin (HSA) and the small molecular weight (SMW) thiols L-cysteine (Cys) and L-homocysteine (h-Cys),[2] the role that SMW thiols play in the translocation of Cd2+ to organs is unclear. To gain insight we have used an approach that involves HPLC (anion exchange chromatography) coupled to a flame atomic absorption spectrometer. To mimic the physiological conditions of human blood plasma a mobile phase with 100 mM NaCl and 5 mM Tris-buffer (pH 7.4) was used and Cd2+ (50 μ g) was injected with Cys (0-1.0 mM) and h-Cys (0-0.4 mM). SMW thiols increased the retention time of Cd which can be rationalised by the formation of negatively charged [Cd(Cys)m(Cl)n]x- and [Cd(h-Cys)m(Cl)n]x- complexes, which may be the Cd-species that are translocated to organs.

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DECISION RULES FOR MULTIANALYTE TESTS. Edgar F. Paski, Analytical Innovations, North Vancouver, BC, Canada (ed@edpaski.com)

Shewhart type control charts are a widely used quality management tool by testing laboratories for ensuring measurement quality. Shewhart decision rules use 3 s (99.74 % confidence interval) for an action limit and 2 s (95% confidence interval) for a warning limit. For a single analyte the likelihood of a Type I inference error (false positive) are 26 in 10,000 for a 3 s action limit and 5 in 100 for a 2 s warning limit. For multianalyte tests such as ICP-OES, ICP-MS, HPLC more than 50 analytes are measured on a single injection of a test solution. The Type I inference error for a 50 analyte test would result in one in eight samples being rejected due to a single analyte exceeding the 3 s action limit. The control chart decision rules to be presented are applicable to multianalyte tests for any number of analytes, they reduce the Type I error to about that of a single element test while evaluating all analytes simultaneously and include trending evaluation.

I056

A CAPILLARY ELECTROPHORESIS ASSAY TO ASSESS THE PREVALENCE AND RISK FACTORS ASSOCIATED WITH IODINE DEFICIENCY IN CANADA. Stellena Mathiaparanam,¹ Adriana Nori de Macedo,^{1,2} Andrew Mente,³ Paul Poirier,⁴ Scott A. Lear,⁵ Andreas Wielgosz,⁶ Koon Teo,³ Salim Yusuf,³ **Philip Britz-McKibbin**¹, ¹Department of Chemistry and Chemical Biology, McMaster University, Hamilton, ON, Canada; ²Departamento de Química, Universidade Federal de Minas Gerais, Belo Horizonte, Minas Gerais, Brazil; ³Population Health Research Institute, McMaster University and Hamilton Health Sciences, Hamilton, ON, Canada; ⁴Faculté de Pharmacie, Institut Universitaire de Cardiologie et de Pneumologie de Québec, Université Laval, Québec City, QC, Canada; ⁵Faculty of Health Sciences, Simon Fraser University, Burnaby and Division of Cardiology, Providence Health Care, Vancouver, BC, Canada; ⁶University of Ottawa Heart Institute, Ottawa, ON, Canada. (BRITZ@MCMASTER.CA)

Iodine is a trace micronutrient that is critical for normal thyroid function and human health. Inadequate dietary intake is associated with cognitive impairment and iodine deficiency disorders in affected populations. Herein, we examined the prevalence of iodine deficiency in Canadian adults based on the analysis of 24 h urine samples collected from 800 participants in the Prospective Urban and Rural Epidemiological (PURE) study. Urinary iodide together with thiocyanate and nitrate were measured using a validated assay based on capillary electrophoresis with UV detection in conjunction with sample self-stacking. Protective/risk factors associated with iodine deficiency were identified using a binary logistic regression model, whereas daily urinary iodine concentration (24 h UIC, µg/L) and urinary iodine excretion (24 h UIE, mg/day) were compared using complementary statistical methods. Overall, our Canadian cohort had adequate iodine status as reflected by a median UIC of 111 mg/L with 11.9% of the population < 50 mg/L as having moderate to severe iodine deficiency. Participants taking iodine supplements, had greater 24 h urine volume, excreted higher urinary sodium, and/or prescribed thyroxine had lower risk for iodine deficiency. Self-reported intake of dairy products was also strongly associated with iodine status. In contrast, participants residing in Quebec City and Vancouver were more susceptible to iodine deficiency than Hamilton or Ottawa. Greater exposure to abundant iodine uptake inhibitors from tobacco smoking and intake of goitrogenic foods was associated with elevated urinary thiocyanate and/or nitrate for participants from Quebec City and Vancouver. Recent public health policies that advocate for salt restriction and lower dairy intake may inadvertently reduce iodine adequacy in Canada.

I057

AN IMPROVED METHOD FOR METHYLMERCURY SPECIATION WITH HPLC-ICP-MS. Kenneth Ong, Caroline Ling and **Aaron Hineman**, PerkinElmer Inc. #6, 501 Rowntree Dairy Rd, Woodbridge, ON, Canada. (<u>Aaron.Hineman@PERKINELMER.COM</u>)

Mercury (Hg) is a toxic element which is present in the environment in several forms from both natural and anthropogenic sources. As it enters water, mercury is consumed fish, where it is metabolized into methylmercury (MeHg), a highly toxic compound. Since marine organisms bio-accumulate Hg, the highest concentrations are found in mature, predatory fish, such as tuna. One of the primary exposure routes to MeHg is through consumption of tuna. Therefore, it is important to monitor the concentration of MeHg is tuna.

Our previous work measured MeHg by HPLC-ICP-MS based on a toluene extraction from the tuna, which resulted in 5 minute separations. Our current work improves this methodology by implementing an aqueous extraction and changing the chromatographic conditions, resulting in separations in 3 minutes. The shorter chromatograms result in taller, narrower peaks, which allows more consistent low-level measurements.

I058

NON-TARGETED SCREENING OF PESTICIDES IN HONEY FROM CANADA USING LC-QTOF-MS. **Shaghig Bilamjian**¹, Lei Tian¹, Caren Akiki¹, Tarun Anumol², Daniel Cuthbertson², Stéphane Bayen¹, ¹Department of Food Science and Agricultural Chemistry, McGill University, Montreal, QC, Canada; ²Agilent Technologies, USA. (shaghig.bilamjian@mcgill.ca)

Throughout their life, honey bees can be exposed to environmental contaminants, including pesticides, via contact and the consumption of contaminated pollen and nectar. Traces of these chemical residues may in turn accumulate in honey and other bee products. In a previous study [1], a method based on direct injection in LC-QTOF-MS was developed for the suspected-target screening of plastic-related chemicals in honey. This novel method relies on the analysis of small amount of honey (0.2 g), requires little sample preparation (<20 min.), and can be easily automated. In this study, a non-targeted workflow was adapted from this earlier approach and evaluated for its capacity to screen for pesticide residues. Good instrumental linearity (1-100 ng/L, r2>0.99) and recoveries (98-105%) were achieved. The workflow was applied to honey samples collected from the Canadian market and the method identification limit (MIL) was determined. The developed workflow could positively identify 42/60 pesticides at a level of 0.1 μ g/g in honey and all pesticides at 1 μ g/g; levels lower or in the range of usual maximum residue limits for pesticides. These results further confirm that the present method is quick, simple and effective, and is effective at screening many classes of contaminants to ensure the quality, authenticity and safety of honey.

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METALS AND FOOD SAFETY, TRENDING TOPICS. **Eve Kroukamp** and Liyan Xing. PerkinElmer Inc. #6, 501 Rowntree Dairy Rd, Woodbridge, ON, Canada. (Eve.Kroukamp@PERKINELMER.COM)

Metals are non-biodegradeable, persistent and once released into the environment can enter into the food chain via various different means. While some metals are of nutritional value and are essential to life, others may be toxic even at very low concentrations and can elicit a variety of adverse effects on plants and animals alike. This talk will cover some of the applications related to metals in foodstuffs which are currently trending at regulatory bodies. Therefore, a few specific topics, such as salt in food and trace metals in baby foods, and the associated analytical challenges, will be discussed in more detail.

I060

ELECTROCHEMICAL HOST-GUEST BIOSENSOR FOR IN BLOOD MEASUREMENTS. **Philippe Dauphin Ducharme**, Département de chimie, Université de Sherbrooke, Sherbrooke, Québec J1K 2R1, Canada (philippe.dauphin.ducharme@usherbrooke.ca)

Amid increasing demands for modernizing cumbersome and laboratory-bound analytical approaches, researchers are developing generalizable electrochemical sensing alternatives for point-of-need applications that are analogous to the glucometer. The overwhelming change in the quality of life of millions provided by the glucometer illustrates how impactful accessing such monitoring device could modernize medicine. Electrochemical sensors are portable, rapid (< 15 min) and afford convenient measurements directly in undiluted complex matrices. To date, however, electrochemical sensors have poor generalizability (i.e, ability to measure different molecules while still relying on a common signal transduction scheme), and thus only a limited number have translated from the laboratory into "real-world" applications. In response, we are developing a generalizable sensing platform using the vast library of host-guest complexes. Hosts present deterministic interactions toward specific classes of guest molecules offering for an unprecedented mean to increase the selectivity in sensing platforms [1]. As a proof-of-concept for this new class of sensors, we decided to use cyclobis(paraquat-p-phenylene) (referred as "blue box") [2], an electroactive host that allows sensing of biologically relevant electron-rich targets such as dopamine, serotonin and acetaminophen. We attached blue box on a surface-attached DNA chain to promote host-guest formation that could be quantified using electrochemistry with response times under a typical doctor-patient interaction and tested the applicability of such sensors to readily deploy in undiluted complex matrices. We envision that in using host-guest chemistry will enable for the development of a new class of electrochemical sensors amenable at the point-of-need.



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POLY-SYNCHRONOUS SOLVENT-BASED SOLID-PHASE NANOEXTRACTION -APPLICATIONS FOR MASS SPECTROMETRY IMAGING. **Malek Hassan**, Jian Yu, Huidong Wang, & Richard Oleschuk. Queen's University, Department of Chemistry, Kingston, ON K7L 3N6, Canada. (malek.hassan@queensu.ca, richard.oleschuk@chem.queensu.ca)

Sample preparation prior to mass spectrometric analysis is a crucial step due to the complex nature of the samples often studied. Mass spectrometry imaging (MSI) in particular requires that the sample be sliced thinly using specialized instrumentation in order to both section and mount the sample for imaging. Sample stamping is a relatively new sample preparation method for MSI that helps reduce sample preparation time and the need for specialized instrumentation. In this work, we use a laser-milled glass substrate coated with medium/long chain fatty acids, which have favorable properties such as high melting points, low polarities, and low toxicities, to stamp and image strawberry and bacterial samples. Different fatty acids (i.e., decanoic, dodecanoic, tetradecanoic, and hexadecanoic acid) are deposited via dip-coating on laser-milled indium tin oxide (ITO) glass slides prior to sample stamping. The acid-coated ITO slides are placed in conformal contact with the tissue sample to enact a localized extraction. The extraction spots are then imaged using a liquid micro-junction surface sampling probe (LMJ-SSP) coupled with electrospray ionization-mass spectrometry (ESI-MS). The LMJ-SSP is manipulated (*x*,*y*,*z*) using a customized 3-D printer chassis, wherein the LMJ-SSP is fitted and subsequently rastered across the sample to produce ion heatmaps.

I062

THE SERUM METABOLOME BY MULTISEGMENT INJECTION-CAPILLARY ELECTROPHORESIS-MASS SPECTROMETRY: A HIGH THROUGHPUT PLATFORM AND STANDARDIZED DATA WORKFLOW FOR LARGE-SCALE EPIDEMIOLOGICAL STUDIES. Zachary Kroezen, Meera Shanmuganathan, Eric Fries, Na-Yung Seoh, Natasia Kurysko, Navneet Kang, Hani Choksi, Philip Britz-McKibbin, McMaster University, 1280 Main Street W., Hamilton, ON L8S 4L8, Canada. (kroezezp@mcmaster.ca)

Metabolomics is a growing field which focuses on quantitative measurements of small molecular intermediates and products of metabolism. There is also growing interest in using metabolomics for better assessment of complex dietary patterns as compared to food frequency questionnaires (FFQs), which are prone to bias while not reflecting contemporary food exposures. However, separation based analytical methods (LC and GC) are time consuming and low throughput. Thus, there is an urgent need for the development of low cost, high throughput yet robust analytical platforms for nontargeted metabolite profiling for biomarker discovery that is amenable to largescale epidemiological studies. Herein, a multiplexed electrophoretic platform based on multisegment injection-capillary electrophoresis-mass spectrometry (MSI-CE-MS) was used to analyze 11745 serum samples from twenty-nine countries around the world. This work will outline a standardized operating protocol and data workflow to ensure reliable and quantitative metabolomic analyses with stringent quality control, including sample workup, preventative maintenance, data pre-processing and batch correction effects due to long-term signal drift. Additionally, reference ranges of serum metabolite concentrations reliably measured in the majority of serum samples are discussed, including metabolites of clinical relevance to nutritional status and cardiovascular risk.

COMPARISON OF MICROWAVE DIGESTION AND DRY-ASHING METHODS FOR TOTAL METAL ANALYSIS OF PINE NEEDLES COLLECTED FROM BUTTE, MONTANA. **Catharina Venter¹**, Kevin Hayes², James Walker², Caleb Marx², Gwen O'Sullivan² and Nausheen Sadiq¹. Mount Royal University, Department of ¹Chemistry and Physics, ²Department of Earth and Environmental Science 4825 Mount Royal Gate SW, Calgary, T3E 6K6, Canada. (cvent117@mtroyal.ca)

The high cost associated with metals analysis in research is driven by the balance of obtaining high-quality data while using optimal reagents. To evaluate cost-effective strategies, we compared acid purity while investigating whether accurate results could still be obtained. This study conducted the analysis of heavy metals (arsenic, lead, iron, cadmium, aluminum, copper, zinc, mercury, selenium, nickel, chromium, manganese, vanadium, cobalt, silver and tin) in dried pine needles collected from Butte Montana, an area surrounded by an open-pit copper mine and one of the largest Superfund sites in the country [1]. Two different digestion methods: dry ashing and microwave digestion were compared and validated with a pine needle certified reference material from High-Purity Standards (CRM-PN-A) using an Agilent 7850 inductively-coupled plasma mass spectrometer (ICP-MS). The digestion methods were repeated for pine needle samples collected from the Butte Montana area. Different grades of Fisher ChemicalTM nitric acid were compared for the microwave digestion method: Certified ACS Plus, TraceMetalTM, and OptimaTM. We intend to further improve digestion methods for future analyses of various organic materials and the impact of the superfund site on the surrounding environment.

[1] C. H. Gammons, & G. A. Icopini, Mine Water Environ 39 (2020) 427-439.

I064

ANALYSIS OF ZINC PEROXIDE NANOPARTICLES DOPED WITH OTHER TRANSITION METALS BY CAPILLARY ELECTROPHORESIS, ELECTROCHEMISTRY AND SPECTROFLUORIMETRY. **Edward P.C. Lai**, Sarah Elab, Eman Elmorsi, Kailai Wang and Kate Yeadon. Carleton University, Department of Chemistry, 1125 Colonel By Drive, Ottawa, ON K1S 5B6, Canada. (edward.lai@carleton.ca)

ZnO₂ nanoparticles, doped with chromium, cobalt, chromium, and other transition metals will be synthesized by a facile sol-gel sonochemical method. These doped nanoparticles will be tested for binding with pharmaceutically active compounds (PACs) in aqueous solution and environmental water samples using capillary electrophoresis, cyclic voltammetry, and spectrofluorimetry. Our objective is to investigate their similarities and differences in chemical properties, towards the development of a selective analytical method for their accurate determination, without interference by PACs and ZnO nanoparticles commonly doped with silver or carbon. The doped ZnO₂ nanoparticles can be applied to de-icing fluids for the formation of an icephobic coating on various substrate materials of relevance to aerospace engineering.

FOOD THERMAL LABELS AS A PATHWAY OF EXPOSURE TO BISPHENOLS AND ANALOGUES. **Ziyun Xu**¹, Lei Tian¹, Lan Liu¹, Cindy Gates Goodyer², Barbara Hales³, Stéphane Bayen¹, ¹Department of Food Science and Agricultural Chemistry, McGill University, Montreal, QC, Canada; ²Department of Pediatrics, McGill University, Montreal, QC, Canada; ³Department of Pharmacology and Therapeutics, McGill University, Montreal, QC, Canada. (ziyun.xu@mail.mcgill.ca)

Bisphenol A and structural bisphenol analogues were used in large quantities as a color developer in thermal paper. Several studies have flagged potential human health risks associated with chronic exposure to BPA and analogues. In this study, we hypothesized chemical migration from thermal label stickers on common packaging could be a dietary source of BPA analogues. To test this hypothesis, multiple packaging samples (n=140) used for fresh food from Montreal were collected to assess the occurrence of bisphenols based on liquid chromatography-mass spectrometry (LC-MS). BPA was not detected in any of the packaging samples (LOD = 1.3 ng/cm²); however, four other bisphenols, BPS, D-8, D-90, TGSA and Pergafast-201, were present in food thermal labels (n=40). Test were then conducted on 24 packaged fish samples to assess the migration of bisphenols from labels into food. Relatively high migration of BPS (up to 1120 ng/g wet weight), D-8 (up to 226 ng/g ww) and PF-201 (up to 137 ng/g ww) were measured in fish wrapped in film with a thermal label (4°C) for 5 days. The migration of D-90 into fish was minimal but detectable (up to 19.6 ng/g ww). This study shows, for the first time, that migration from thermal label stickers is a major source of bisphenols in our diet; further detailed risk assessment is required.

I066

ANALYTICAL CHALLENGES AND ADVANCES IN LONGITUDINAL LC-MS LIPIDOMICS STUDIES. Lise Cougnaud, Oluwatosin Kuteyi, **Dajana Vuckovic**. Concordia University, Department of Chemistry and Biochemistry, 7141 Sherbrooke Street West, Montreal, QC H4B 1R6, Canada. (dajana.vuckovic@concordia.ca)

In this talk, I will discuss several key considerations and challenges in LC-MS lipidomics and summarize our main strategies to improve lipid measurements: increasing lipid coverage, capturing unstable lipids for their accurate measurement, reducing the invasiveness of blood sampling and the international standardization/harmonization. Specifically, I will present two studies on lipid stability, and describe the potential of lipase inhibitors or solid-phase microextraction to stabilize unstable lipids in biospecimens. I will further compare and contrast our recent solid-phase extraction and in vivo solid-phase microextraction approaches for the accurate measurement of eicosanoids and related lipids suitable for longitudinal studies. These new analytical methods can offer new insights into lipid biology and facilitate biomarker discovery and validation. To demonstrate this, I will briefly present longitudinal oxylipin profiling data in plasma collected over 6-weeks in ApoE-mice (n= 108) and show early disturbances in linoleic acid and omega-3 fatty acid pathways.

ELECTROCHEMICAL AND IN SITU FTIR SPECTROSCOPIC STUDY OF CO₂ REDUCTION. **Aicheng Chen**, University of Guelph, Department of Chemistry, 50 Stone Road East, Guelph, ON N1L 1P1, Canada. (<u>aicheng@uoguelph.ca</u>)

There is a growing interest in developing high-performance catalysts for the electrochemical reduction of carbon dioxide (CO₂) to address the increasingly serious impacts of global climate change. In this talk, we report on the design of advanced three-dimensional (3D) nanomaterials (e.g., nanoporous gold, Cu nanodendrites and Co nanodendrites) for the efficient electrochemical reduction of CO₂. The morphology, composition and structure of the synthesized 3D nanomaterials were characterized with various imaging and spectroscopic techniques, including FE-SEM, XRD, EDX and XPS. The effects of an applied potential on the electrochemical reduction of CO₂ were investigated using various electrochemical methods. The products generated from the CO₂ electrochemical reduction were identified by gas chromatography and nuclear magnetic resonance (NMR) spectroscopy. The kinetics of the CO₂ reduction reaction at the 3D nanomaterials was further studied using *in situ* electrochemical Fourier transform infrared (FTIR) spectroscopy. The critical roles of nanostructured surfaces in the electrochemical reduction of CO₂ are discussed.

I068 P

USING FLUORESCENT CARBON DOTS FOR DETECTION OF NITROTYROSINE. Nayomi Camilus¹, Rafik Naccache², Sanela Martic¹. ¹Trent University, Department of Forensic Science, 1600 W Bank Dr, Peterborough, ON K9L 0G2, Canada. ²Concordia University, Department of Chemistry and Biochemistry, 1455 Boulevard de Maisonneuve O, Montréal, QC H3G 1M8, Canada. (nayomicamilus@trentu.ca)

Nitrotyrosine is linked to diseases, including neurodegeneration, making its detection have diagnostic value. Nanomaterials have a wide range of applications in sensors, catalysis, environmental monitoring, and are valuable sensing materials. Carbon dots (CDs), one type of nanomaterials, exhibit low toxicity and desirable photophysical properties for biosensing applications. The photophysical properties of dual-fluorescing CDs were tested using UV-vis and fluorescence spectroscopy. The fluorescent assay was highly selective for nitrotyrosine. The fluorescence intensity was quenched by nitrotyrosine, even in the presence of tyrosine and phosphotyrosine. In the 0.005-0.304 mM concentration range of nitrotyrosine, significant fluorescence quenching (about 55%) was observed. Interference studies indicate that the CDs are ideally suited for detection of nTyr even in the presence of other biologically relevant molecules like amino acids, nucleosides and metal ions. The detection of nitrotyrosine using CDs was tested in biological matrices including diluted artificial saliva, bovine serum albumin and equine serum and the results show that the fluorescence CD-based assay may be used in complex mixtures to mimic the biological fluids. In conclusion, data indicate that water soluble CDs are ideal for biosensing applications.

ROLE OF GUANOSINE ON THE PHOTOPHYSICAL PROPERTIES OF CARBON DOTS. **Nayomi Camilus**¹, Finlay Dingman Peterson¹, Musonda Mitti¹, Rafik Naccache², Sanela Martic^{1*}. ¹Trent University, Department of Forensic Science, Environmental and Life Sciences Program, 1600 W Bank Dr, Peterborough, ON K9L 0G2, Canada. ²Concordia University, Department of Chemistry and Biochemistry, 1455 Boulevard de Maisonneuve O, Montréal, QC H3G 1M8, Canada. (<u>nayomicamilus@trentu.ca; sanelamartic@trentu.ca</u>)

Carbon dots (CDs) are carbon-based nanomaterials with versatile optical properties, high photostability and solubility. CDs have excellent biocompatibility as well as low cytotoxicity. Their photoluminescent properties are dependent on size, excitation wavelength, and functionalization. Hence, CDs are ideally suited as optical probes for a variety of biomolecules. In this project, the photophysical properties of CDs were evaluated in the presence or absence of a nucleoside, specifically, guanosine, by using UV-vis and fluorescence spectroscopy. Dual-fluorescence emission was quenched in the presence of guanosine, but the quenching was highly dependent on the excitation wavelengths used. The fluorescence quenching assay, a linear response was observed at low concentrations in the micromolar range and an upward curvature at higher concentrations indicating both dynamic and static quenching processes. Data were compared to a traditional quencher (Cu(II) metal ions). The data indicate that CDs are valuable biological probes for the detection of nucleic acid building blocks.

I070

MONITORING UPTAKE OF BINDING-MEDIATED PROTEIN CORONA BY LIVING CELLS USING ICP-MS. Jinjun Wu, Hanyong Peng, X. Chris Le, **Hongquan Zhang**. Department of Laboratory Medicine and Pathology, University of Alberta, Edmonton, AB T6G 2G3, Canada (hongquan@ualberta.ca)

Nanoparticles (NPs) are known to adsorb proteins non-specifically, forming a multi-layered protein coating called "protein corona". However, the non-specific nature of protein corona formation makes it difficult to deliver specific proteins for therapeutic applications. Herein, we report on the construction of a new type of protein corona, termed binding-mediated protein corona. It uses affinity ligands pre-conjugated onto NPs to assemble specific payload proteins into a uniform monolayer and serve in a spatial role to avoid irreversible binding of the payload proteins directly with the core of the NPs. We used ICP-MS to study the cellular uptake of the binding-mediated protein corona and found its cellular uptake can be rationally modulated through control of density of the protein outer layer. We observed that the cellular uptake of binding-mediated protein corona. Successful gene editing in human cell lines (Hela and HEK293) demonstrates the efficient delivery, high stability, low cytotoxicity, and well-controlled activity of the Cas9-guide RNA ribonucleoprotein.

BIOPHYSICAL CHARACTERIZATION OF GELSOLIN PEPTIDES: AGGREGATION AND INHIBITION. **Carlos Quintero Arias**¹, Sanela Martic^{1,2}, ¹Department of Forensic Science, ²Environmental and Life Sciences Program, Trent University, Peterborough, ON, Canada. (cquinteroarias@trentu.ca)

Gelsolin is an actin-binding protein responsible for remodelling the cytoskeleton [1,2]. Point mutations have been associated with the aberrant proteolytic cleavage and formation of amyloid fibrils; the resulting fragments systematically deposit within the organs to promote cell death [2]. Gelsolin protein aggregates are also capable of propagating their abnormal confirmation in a prion-like manner [2]. Despite the detrimental effects of Gelsolin mutation, the mechanism of amyloid formation is understudied. Herein, we used various biophysical and bioanalytical methodologies to investigate the aggregation propensities, inhibition, and morphology of wild-type Gelsolin peptides (187-193) and their associated mutants. We also explored the seeding reaction of Gelsolin to monitor the propagation of the diseased state. Spectroscopic and microscopic studies allowed for the identification and visualization of aggregates and amyloid formation. All CFILDL-containing peptides were prone to aggregation, which may be inhibited and reversed by small molecules. Certain mutations had a pronounced effect on aggregation propensities pointing to their biological relevance.

[1] M Ahmad, J Esposto, C Golec, C Wu, S Martic-Milne, Mol Cell Biochem (2021) 476 2393–2408.

[2] JP Solomon, LJ Page, WE Balch, JW Kelly. Crit Rev Biochem Mol Biol (2012) 47 282–296.

I072

PATTERNED WETTABILITY AND CONDUCTIVE TOUCH FOR IMPROVED TISSUE PROFILING WITH LIQUID MICROJUNCTION SURFACE SAMPLING **Richard Oleschuk**, Matthias Hermann, Haidy Metwally, Jian Yu, Rory McEwan, Phillip Hillen, Ben Tucker, Kasia Donovan, Jess Deng, Queen's University, Department of Chemistry, 90 Bader Lane, Kingston, ON K7L 3N6, Canada. (richard.oleschuk@chem.queensu.ca)

Mass spectrometric profiling typically requires significant sample preparation prior to analysis. Ambient ionization methods have further reduced sample preparation requirements however most still require the sample to be embedded and/or sliced extremely thin, coated with matrix and/or dehydrated/dewaxed prior to MS analysis. We have employed (super)hydrophobic coatings combined with laser micromachining and plasma etching to produce surface energy traps (SETs). These hydrophilic regions spontaneously capture droplets through a process termed discontinuous de-wetting (DDW). DDW can be used to accurately deposit and array thousands of droplets with no need for traditional pipetting or spotting. The droplets are contacted with a tissue surface to spatially extract analytes for MS profiling. In addition, a Liquid Micro Junction-Surface Sampling Probe (LMJ-SSP) is used to interrogate tissue samples either indirectly using the droplet array or directly on the tissue surface. We have a developed a conductance feedback approach that precisely positions the probe by sensing fluidic contact with the sample through a conductive feedback approach. As a result, the probe can profile tissue with large variations in thickness/shape. We will show profiling tissue examples including resected human kidney (intratumoral hemorrhage) and breast tumor specimens 1-4 mm in thickness using the automated LMJ-SSP system.
MONITORING ELECTROCHEMICAL OXIDATION REACTION AND PRODUCT FORMATION FROM BULKY PHENOLS. **Abigail Boon**, Sanela Martic. Department of Forensic Science, Environmental and Life Sciences Program, Trent University 1600 West Bank Drive, Peterborough, Canada. (abigailoon@trentu.ca, sanelamartic@trentu.ca)

Phenolic compounds have various applications in the food, agriculture, and automotive industries. Due to their widespread use they find their way into the environment which leads to adverse effects on ecosystems. Recently the selective electrochemical oxidation of bulky phenols was accomplished and compared to chemical oxidation [1]. Using cyclic voltammetry, 2,6-di-tert-butylphenol (DTBP) was transformed into 3,5,3',5'-tetra-tert-butyl-4,4'-diphenoquinone (TTBDQ), a highly absorbing species which was characterized through X-ray crystal diffraction [1]. The electrosynthesis was monitored using spectroscopy, and reaction parameters, such as applied potential and substrate concentration, among others, were optimized, and reaction compared to traditional chemical oxidation. Several bulky phenol analogues were also compared to determine selectivity of electrochemical synthesis. Data show that unlike traditional organic oxidation leads to substrate and product selectivity.

[1] N. Zabik, C. Virca, T. McCormick, S. Martic-Milne. J. Phys. Chem. B. 120 (2016) 8914-8924

I074

THE VALIDATION AND ANALYSIS OF DIETARY URINE BIOMARKERS IN RELATION TO CHRONIC DISEASE RISK. Biban Gill¹, **Vanessa Martinez¹**, Erick Helmeczi¹, Stellena Mathiaparanam¹, Sathish Thirunavukkarasu², Gui Paré², Salim Yusuf², Koon Teo², Philip Britz-McKibbin¹, ¹Department of Chemistry & Chemical Biology, McMaster University, Hamilton, ON, Canada; ²Population Health Research Institute, McMaster University, Hamilton, ON, Canada. (martinezvan42@gmail.com)

Current methods used to examine diet involve food frequency questionnaires (FFQs), which are prone to bias and misreporting. Although there is growing interest in using dietary biomarkers as objective indicators of food intake, limited studies have validated their generalizability in diverse populations. Herein, we applied a nontargeted metabolomics workflow by multisegment-injection capillary electrophoresis-mass spectrometry (MSI-CE-MS) with multivariate and univariate statistical analysis methods for identifying robust dietary biomarkers in urine samples from participants in the Prospective Urban and Rural Epidemiology (PURE) study. A total of 116 urinary metabolites were quantified by MSI-CE-MS (CV < 30%) from participants (n=1000) from 14 countries with varying income status and compared to FFQs (n=60). Overall, eight top-ranked candidates were considered robust dietary biomarkers based on their dose response, plausibility, and generalizability. Urinary trigonelline, proline betaine, carnitine, 3-methylhistidine, saccharin, and acesulfame-K were replicated across a large multiethnic cohort reflecting variable and regional dietary patterns associated with the consumption of coffee, fruits and vegetables, red meat, total sugar, and processed foods. Our study highlights that a panel of urinary dietary biomarkers of food intake may provide more reliable assessment of dietary patterns globally while providing new insights into diet quality and chronic disease risk in diverse populations.

A HIGH-THROUGHOUT PLATFORM FOR THE RAPID SCREENING OF VITAMIN D STATUS BY DIRECT INFUSION-MS/MS. Erick Helmeczi¹, Eric Fries¹, Lauren Perry², Karen Choong², Katie O'Hearn³, Dayre McNally^{3,4}, and Philip Britz-McKibbin¹, ¹Department of Chemistry and Chemical Biology, and ²Department of Pediatrics and Critical Care, McMaster University, Hamilton, ON, Canada; ³Children's Hospital of Eastern Ontario Research Institute, Ottawa, ON, Canada; ⁴Department of Pediatrics, Children's Hospital of Eastern Ontario, University of Ottawa, Ottawa, ON, Canada. (HELMECWE@McMaster.ca)

Vitamin D is an important fat-soluble prohormone with pleiotropic effects on human health, such as immunomodulation of the innate and adaptive immune system. There is an unmet clinical need for a rapid screening platform for 25-hydroxyvitamin D (25OH-D) determination without chromatographic separation that offers better precision and accuracy than immunoassays. Here, we introduce a high-throughput method for assessing vitamin D status from blood specimens based on direct infusion-MS/MS (DI-MS/MS) following click derivatization using 2-nitrosopyridine. We developed an optimized liquid-phase extraction protocol to minimize ion suppression when directly infusing serum or plasma extracts via a capillary electrophoresis system for quantitative determination of 25OH-D. Acceptable reproducibility (mean coefficient of variation = 10.9%, n =412), recovery (mean = 102% at 15, 30, and 45 nmol/l), and linearity ($R^2 > 0.998$) were achieved for 25OH-D with lower detection limits (limit of detection ~1.2 nmol/l, $S/N \sim 3$), greater throughput (\sim 3 min/sample), and less bias than a commercial chemiluminescence immunoassay prone to batch effects. There was mutual agreement in 25OH-D concentrations from reference blood samples measured by DI-MS/MS as compared with LC-MS/MS (mean bias = 7.8%, n = 18). We also demonstrate that this method could reduce immunoassay misclassification of vitamin D deficiency in a cohort of critically ill children (n = 30). In conclusion, DI-MS/MS offers a viable alternative to LC-MS/MS for assessment of vitamin D status in support of large-scale studies in nutritional epidemiology as well as clinical trials to rapidly screen individual patients who may benefit from vitamin D supplementation.

I076

NANOPARTICLES SIZE ANALYSIS BY SEDIMENTATION AND CHARGE ANALYSIS BY MICROFLUIDIC ELECTROPHORESIS USING A 3D-PRINTING DEVICE. **Edward P.C.** Lai. Carleton University, Department of Chemistry, 1125 Colonel By Drive, Ottawa, ON K1S 5B6, Canada. (edward.lai@carleton.ca)

Transition metal oxide nanoparticles, doped with chromium, cobalt or chromium, will be analyzed using an apparatus of novel design. The nanoparticles are separated first by size based on sedimentation under gravity and then by charge based on electrophoresis through parallel microfluidic channels. They will be analyzed again after binding with pharmaceutically active compounds (PACs) in aqueous solution. Detection by both conductivity and light scattering will be evaluated in addition to UV absorption and fluorescence emission. Our objective is to develop a device that can be manufactured by 3D printing for screening analysis of nanoparticles in environmental water samples contaminated by soil, without prior membrane filtration.

DIRECT ANALYSIS OF TRACE ELEMENTS IN SEAWATER USING ICP-MS WITH VERSATILE REACTION MODES. Liyan Xing and Chady Stephan; PerkinElmer Inc., Woodbridge, ON, Canada. (LIYAN.XING@PERKINELMER.COM)

The determination of trace elements in seawater is usually considered to be challenging for ICP-MS due to the high total dissolved solids (TDS) in this matrix. The high concentrations of matrix components in seawater, such as sodium, magnesium, and chloride ions, may form polyatomic spectral interferences and complicate the determination of elements, such as As, Co, V, Zn, Cu, and Fe. Even for elements like Cd, Sn, and Pb that are less affected by spectral interferences, the low ppt concentrations in seawater make them difficult to determine with good accuracy and precision. Both NexION® 2000 ICP-MS and NexION 5000 Multi-Quadrupole ICP-MS are equipped with Universal Cell Technical (UCT), which allows for samples to be run in Standard, Collision with Kinetic Energy Discrimination (KED), and Reaction with Dynamic Bandpass Tuning (DBT) modes. Here, we reported a direct analysis procedure using online dilution with a combination of liquid dilution and gas dilution. A variety of reaction/collision gas modes were applied to demonstrate the versatility of the method. The accuracy of the method is validated by the analysis of seawater certified reference materials NASS-7, CASS-6, and MX014.

I078 P

IDENTIFICATION OF COPPER, IRON AND ZINC **METALLOPROTEINS** IN ERYTHROCYTE CYTOSOL BY SIZE-EXCLUSION CHROMATOGRAPHY-INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROSCOPY. Marvam Doroudian and Jürgen Gailer, Department of Chemistry, University of Calgary, Calgary, Canada. (maryam.doroudian@ucalgary.ca)

The chronic exposure of humans to toxic metal(loid)s is associated with adverse health effects the biomolecular basis of which is poorly understood. Endogenous metalloproteins represent an underutilized class of biomolecules which can provide important insight into the dyshomeostasis of essential elements, but their determination in complex biological fluids (e.g., blood plasma, erythrocyte cytosol) is challenging. To overcome these challenges bioanalytical techniques involving the hyphenation of high-resolution separation techniques with sensitive multielement specific detection techniques can be employed. To this end, the rapid analysis (<26 min) of erythrocyte cytosol for Cu, Fe, and Zn-containing metalloproteins by size-exclusion chromatography (SEC; Superdex 75, 30 x 1.0 cm ID) coupled online to an inductively coupled plasma atomic emission spectrometer (ICP-AES) has been reported [1]. To identify individual metalloproteins we have analyzed erythrocyte cytosol spiked with highly pure metalloproteins. To date, this approach has allowed us to unequivocally identify hemoglobin (64.5 kDa) and carbonic anhydrase (29.2 kDa) and experiments are underway to identify Cu, Zn superoxide dismutase (32 kDa) and catalase (57 kDa).

[1] M.A. Gibson, S. Sarpong-Kumankomah, S. Nehzati, G.N. George, J. Gailer, Remarkable differences in the biochemical fate of Cd2+, Hg2+, CH3Hg+ and thimerosal in red blood cell lysate, Metallomics. 9 (2017) 1060–1072.

I077

MAKING 3D PAPER-BASED ANALYTICAL DEVICE ON A SINGLE-SHEET OF 2D FILTER PAPER. Lishen Zhang; Daniel O. Reddy; Timothy T. Salomons; Richard Oleschuk.* Queen's University, Department of Chemistry, 90 Bader Lane, Kingston, ON K7L 3N6, Canada. (lishen.zhang@queensu.ca; richard.oleschuk@chem.queensu.ca)

Microfluidic paper based analytical devices (μ PADs) have been used as substrates for a number of assays. However, a simple method to create μ PADs with complex and precise designs has remained challenging. Herein, we present a new method to create μ PADs with fine resolution and diverse patterns. Filter paper was rendered hydrophobic using a simple organosilane treatment, followed by laser milling, which transforms the laser-milled areas from hydrophobic to hydrophilic. The created channel widths range from tens of micrometers to centimeters. Moreover, the milled depth is a controllable parameter by modulating the laser power. At a given laser power, a hole can be cut through the filter paper, thus turning the filter paper into a 3D device by conducting the liquid flow from top-to-bottom side via capillary forces and gravity. Such a strategy enables the creation of new 3D microfluidic devices without any additional filling materials to transport solution between layers. Then, a colorimetric test was performed to assess the feasibility of this device for real-world applications.

I080

DIRECT ANALYSIS OF INSECT-BASED PROTEINS USING ELECTROTHERMAL VAPORIZATION COUPLED WITH INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETRY. **Yulianna Holowati**¹, Diane Beauchemin¹, Kelly Leblanc², and Zoltan Mester ², ¹Queen's University, Department of Chemistry, 90 Bader Lane, Kingston, ON K7L 3N6, Canada; ²National Research Council of Canada, Ottawa, ON, Canada (diane.beauchemin@queensu.ca)

Insects are becoming a popular and attractive alternative to conventional animal-based proteins. Yet, there is currently little information available on their elemental content. Furthermore, there are currently no methods that exist for the direct analysis of insect-based proteins. Food samples are most commonly analyzed using either microwave-assisted or closed vessel acid digestion which may be time-consuming and lead to contamination and/or loss of analytes. Therefore, a direct multi-element analysis method was developed using electrothermal vaporization (ETV) coupled with inductively coupled plasma optical emission spectrometry (ICPOES). External calibration was performed using increasing amounts of NIST 1586b (rice flour) standard reference material. With 5 mL/min CF₄ reaction gas added to the carrier gas to increase analyte volatility and internal standardization using the Ar 404.442 nm emission line to compensate for sample loading effects on the plasma, the measured concentrations of Co, K, and S agreed with those obtained by ICP mass spectrometry (ICPMS) using standard addition following microwave digestion. When high purity N₂ was added to the plasma central channel using a sheathing device to increase plasma robustness, detection limit, and sensitivity, the concentrations of Cd, Co, Fe, K, Pb, and Zn agreed with those obtained by ICPMS. Therefore, this method allows for the direct multi-elemental analysis of insect-based proteins without the need for sample preparation.

USING SPECTROSCOPY TO MANAGE PHOSPHORUS IN AGRICULTURAL SOILS **Barbara J. Cade-Menun**. Agriculture & Agri-Food Canada, Swift Current Research and Development Centre, Box 1030, Swift Current, SK, S9H 3X2, Canada. (<u>barbara.cade-menun@agr.gc.ca</u>)

Phosphorus (P) is an element of concern in agricultural and environmental studies. An essential nutrient for all organisms, crop production relies on adequate soil P concentrations and may require fertilization. However, fertilizer P in soil can become unavailable to plants over time, and excess P can be transferred to water, triggering growth of harmful algae. Managing P to optimize crop yields while minimizing P losses requires a detailed understanding of P biogeochemistry. Historically, soil P chemistry has focused on phosphate, using simple colorimetric methods. However, it has become widely recognized that a full understanding of P cycling requires the identification of P forms by advanced techniques such as ³¹P nuclear magnetic resonance (P-NMR) and P X-ray Absorption Near-Edge Structure (P-XANES) spectroscopy, and the subsequent linking of these P forms to the chemical and biological factors controlling their cycling. Following an introductory overview of P forms and cycling in soils and environmental samples, results will be presented from studies using P-NMR and P-XANES to investigate P cycling in agricultural soils, including different agriculture land uses; legacy fertilizer P availability in soils; the effects of soil acidification on P cycling; and P forms in snowmelt runoff from agricultural lands.

I082

DEVELOPMENT OF A RAPID UV-ACTIVATED TOTAL OXIDIZABLE PRECURSOR (TOP) ASSAY FOR IMPROVED QUANTIFICATION OF PER - AND POLYFLUOROALKYL SUBSTANCES (PFAS) IN AQUEOUS FILM FORMING FOAM (AFFF). **Natalia O'Connor**¹, David Patch¹, Iris Koch¹, Kela Weber¹, Jinxia Liu², ¹Environmental Sciences Group, Department of Chemistry and Chemical Engineering, Royal Military College of Canada, Kingston, ON, K7K 7B4, Canada. ²Department of Civil Engineering, McGill University, 845 Sherbrook St., Montreal, QC, H3A 0G4, Canada. (natalia.oconnor@rmc-cmr.ca)

PFAS are a large class of synthetic aliphatic organofluorine compounds that are ubiquitious, recalcitrant, and linked to a growing number of toxicological effects. A large majority of PFAS cannot be quantified using standard liquid chromatography-mass spectrometry methods. To address this issue, the total oxidizable precursor (TOP) assay was developed by Houtz and Sedlak (2012) to convert unquantifiable PFAS into quantifiable PFAS end-products. This study focuses on the development of a rapid UV-activated TOP assay to allow for complete conversion of PFAS precursors. The recovery and conversion of 6:2 fluorotelomer sulfonate (6:2 FTS) (a surrogate for PFAS precursors) was examined following either heat or UV-activated TOP assay. The use of UV activation resulted in 99% molar recovery of the generated perfluorocarboxylic acids (PFCAs), compared to 62% molar recovery with heat activation. UV-activated TOP assay continued to demonstrate higher PFAS recoveries compared to heat activation when applied to more complex samples, including aqueous film forming foams. Overall, the UV-activated TOP method proved to be faster and resulted in higher recoveries compared to a standard thermal-activated TOP assay. The improved method can be used to contribute to a more comprehensive understanding of total PFAS load in samples.

MASS SPECTROMETRY BASED STRATEGY FOR IMMUNOPEPTIDOME ANALYSIS Rui Chen, Kelly M. Fulton, Anh Tran, Diana Duque, Susan M. Twine, **Jianjun Li**, Human Health Therapeutics Research Centre, National Research Council Canada, 100 Sussex Drive, Ottawa, ON K1A 0R6, Canada. (jianjun.li@nrc-cnrc.gc.ca)

Immunopeptidomics is the study of the peptides presented by human leukocyte antigen (HLA) molecules on the cell surface that form the major histocompatibility complex (MHC). The immunopeptidome could be utilized for identifying antigenic peptides for cancer immunotherapy and the prevention of infectious diseases. To facilitate the discovery of tumor neoantigens and viral antigens, an advanced immunopeptidomics platform was using a combination of immunoaffinity purification of the HLA molecules and liquid chromatography-tandem mass spectrometry (LC-MS/MS). In this presentation, we present the development and application of our immunopeptidomics platform. For neoantigen discovery, we developed a novel chemical derivatization strategy that combines the analysis of native, dimethylated, and alkylamidated peptides by LC-MS/MS to expand the coverage of immunopeptidome. Our results revealed that dimethylation increases hydrophobicity and ionization efficiency of MHC class I peptides, while alkylamidation significantly improves the fragmentation by producing more y-ions during MS/MS fragmentation. For viral antigen presentation, we developed an integrated immunopeptidomics and proteomics strategy for studying SARS-CoV-2 infection. The glycosylation analysis of HLA proteins from the elution and flow-through of immunoprecipitation revealed that the synthesis and degradation of HLA protein was affected by SARS-CoV-2 infection. The results suggested that viral infection led to the disruption of antigen presentation.

I084

HYPERSPECTRAL VISUALIZATION BASED REAL-TIME MICROBIAL MASS SPECTROMETRY IMAGING. **Jian Yu**, Haidy Metwally, Jennifer Kolwich, Hailey A. Tomm, Avena C. Ross, Richard D. Oleschuk^{*}. Department of Chemistry, Queen's University, Kingston, Ontario K7L 3N6, Canada. (jian.yu@queensu.ca, richard.oleschuk@chem.queensu.ca)

Mass spectrometry imaging (MSI) has been wildly adopted for its exceptional performance in combining spatial and molecular information. However, dynamic monitoring of bacteria growth has been out of reach as sample preparation usually alters the sample. We have been examining the use of liquid micro-junction surface sampling probe (LMJ-SSP) to directly sample and image *Pseudoalteromonas* colonies without sample preparation. Real-time sampling on different strains is performed, while hyperspectral visualization method is employed to give a straightforward interpretation of their dynamic interaction based on principal component analysis (PCA). RGB color-coding translated from PCA score is used to find corresponding m/z values. One can witness the appearance/expression of prodiginine and tambjamine compounds in parallel with the growth of *P. rubra* and *P.tunicata*. Different growth rates of different strains, observed through the expansion of colony boundaries, are easily observed in hyperspectral images. The m/z values of metabolites other than prodiginine and tambjamine can be spotted in spectra at later stages of the life circle. Although not all the novel m/z have yet been identified, different spectra typically signify a dynamic metabolism status. Here, hyperspectral visualization-based *in-situ* MSI is demonstrated to be valid for the dynamic monitoring of bacteria growth.

FLOW-THROUGH SUBWAVELENGHT NANOAPERTURES FOR THE DETECTION OF GAS-PHASE ANALYTES. **Juan Gomez-Cruz**¹, Swapnil Daxini², Jack A. Barnes³, Hans-Peter Loock², Carlos Escobedo¹, ¹ Department of Chemical Engineering, Queen's University, Kingston, ON K7L 3N6, Canada; ² Department of Chemistry, University of Victoria, Victoria, BC, V8W 2Y2, Canada; ⁴ Department of Chemistry, Queen's University, 90 Bader Lane, Kingston, ON K7L 3N6, Canada. (j.gomezcruz@queensu.ca)

Volatile organic compounds (VOCs) are employed in the production of plastics and paints. Prolonged exposure to these compounds has been demonstrated to have serious health side effects, making the development of real-time sensors necessary for detecting and quantifying ambient VOC concentrations. VOCs are readily absorbed by silicone-based inorganic polymers, such as polydimethylsiloxane (PDMS), due to their strong affinity to non-polar molecules. Exposure of PDMS to VOCs causes physical and chemical changes. It also modifies the optical properties by altering the refractive index (RI), depending on the VOC optical properties, solvent-polymer affinity, and sorption kinetics. Near-surface RI changes can be detected with high sensitivity using nanoplasmonic-based sensing techniques, specifically with spectroscopic surface plasmon resonance (SPR). In this work, we present a VOC gas sensor based on PDMS-coated hexagonal flow-through metallic nanohole arrays (NHAs) capable of measuring the uptake and release of VOCs into the PDMS. The sensor measures the change in RI of PDMS by tracking the spectral shift of the plasmonic signal in transmission mode. The analyte concentration of VOCs such as Xylenes within the film was calculated using the experimentally obtained RI and thickness changes into a mathematical model of the diffusion kinetics of the solvent flow.

I086

ARSENIC SPECIATION IN CANADIAN PEAT POGS. Isabelle Hébert, Dr. Iris Koch, Blaire Coffey, David Patch. Royal Military College of Canada, Environmental Sciences Group, 12 Verité Avenue, Kingston, K7K 7B4, ON, Canada. (19ih6@queensu.ca)

Peat bogs are a type of wetland ecosystem characterized by wet and poorly drained peat-rich soil largely consisting of partially decayed plant matter. Arsenic, known to be historically toxic, has been found to accumulate in peat bogs near sites where anthropogenic activities occur. However, arsenic has numerous chemical forms, all varying in toxicity in the environment. A previous study of peat bogs in an area impacted by aerial deposition of arsenic has shown large proportions of a non-toxic form of arsenic, called arsenobetaine [1]. The objective of this project is to analyze Canadian peat bog samples taken from Ottawa, ON, and areas in Ontario's Ring of Fire to determine the species of arsenic present, and to better understand arsenic's potential risk, toxic mechanisms, transformation, and distribution in the environment. Total arsenic values for Ring of Fire samples were obtained using x-ray fluorescence (XRF) and range from 380 to 6000 mg/kg. The chemical forms of arsenic in acidic extracts of these samples (containing 0.94 to 45 mg/L) will be determined by high performance liquid chromatography - inductively coupled plasma mass spectrometry (HPLC-ICPMS) and reported, along with their significance.

[1] Mikutta and Rothwell, Environ. Sci. Technol. (2016) 50, 4314–4323

STATISTICAL EVALUATION OF THE UNCERTAINTIES IN THE CHARACTERIZATION OF SOUTH AFRICAN LOW-GRADE GOLD MINE TAILINGS USING ICP-OES. **Kedibone Mashale^{1,2}**, James Sehata¹, Luke Chimuka² and James Tshilongo¹, ¹Analytical Chemistry Division, Mintek, 200 Malibongwe Drive, Praegville, 2194, South Africa; ²Molecular Sciences Institute, School of Chemistry, University of the Witwatersrand, Braamfontein, 2000, South Africa. (KediboneMa@mintek.co.za)

Accuracy and method confidence in analytical chemistry continues to be of utmost importance, however, these are often overlooked in the initial stages of analysis, such as in sample characterization. It is rather known that the outcome of the characterization techniques contributes highly to the subsequent method development. Therefore, the aim of this study was to evaluate the uncertainties associated with the characterization of geological samples. This involved the quantification of common base metals such as iron, arsenic, nickel and sulphur in a low-grade gold tailing sample, which are known to be interferents in analytical methods subsequent to characterization. Fusion, acid digestion and XRF coupled to instrumental analysis using an ICP-OES were used for elemental and oxide analysis. Characterization of the sample by the mentioned methods showed the presence base metals and minerals typical of mining samples. Uncertainty evaluation was then carried out on the fusion, XRF, acid digestion analysis at a coverage factor of 2. The measurement result in its full form for iron through fusion, XRF and acid digestion was found to be $3.35\pm0.026\%$, $3.81\pm0.023\%$ and $3.69\pm0.020\%$, respectively and that of arsenic were 87.82±11.12 mg/kg and 84.75±8.29 mg/kg for fusion and acid digestion, respectively. The three methods can therefore be associated with high statistical confidence and therefore eases decision making.

I088

HOW DO YOU CONDUCT ARSENIC SPECIATION ANALYSIS WHEN YOUR ICP-MS ISN'T WORKING? **Iris Koch**, Blaire Coffey, David Patch, Natalia O'Connor, Jennifer Scott, Kela Weber. Royal Military College of Canada, Department of Chemistry and Chemical Engineering, 12 Verité Ave, 17000 Station Forces, Kingston, ON K7K 7B4, Canada. (koch-i@rmc.ca)

Speciation analysis is crucial in understanding the many chemical forms of arsenic, and their associated fates and toxicities. However, most conventional arsenic speciation analysis relies on the use of an inductively coupled plasma (ICP) technique (most often with mass spectrometry, MS, for low detection limits) as a detector following a separation technique, most commonly high-performance liquid chromatography (HPLC). A little-discussed problem is presented by aging ICP-MS instruments, which can fail and prevent routine speciation analysis. The presentation will summarize some of the alternative methods that our group has explored, including HPLC-MS, solid-phase extraction x-ray fluorescence, x-ray absorption near edge structure, and other techniques.

I089 P

BIO-ACCESSIBILITY STUDIES OF BLACK SOLDIER FLY LARVAE USING ON-LINE CONTINUOUS LEACHING METHOD COUPLED WITH INDUCTIVELY COUPLD PLASMA MASS SPECTROMETRY. **Qiqi Zhang¹**, Cameron Pytyck¹, Zoltan Mester², Diane Beauchemin¹. ¹Queen's University, Department of Chemistry, 90 Bader Lane, Kingston, ON K7L 3N6, Canada; ²National Research Council of Canada, Ottawa, ON, Canada. (<u>17qz@queensu.ca</u>)

Compared to other edible insects, black soldier fly (Hermetia illucens) larvae (BSFL) is a promising alternative protein source because it can convert various waste into its nutrient body mass. Heavy metal concentration contained in food does not represent the amount dissolved in our gastrointestinal tract (bio-accessibility) and the fraction reaching to the blood stream (bioavailability) equals to the bio-accessible amount in the worst case scenario only. We applied the previously established continuous on-line leaching method to determine bio-accessible amount of Ni, As, Cr, Cd, Pb, Hg, and Se in BSFL. We used a high performance liquid chromatography pump to sequentially pump artificial saliva, gastric, and intestinal juices into a mini-column containing the sample (with the juices and columns maintained at 37°C) while continuously analyzing the effluent by inductively coupled plasma mass spectrometry. This work provides real-time leaching profiles that include useful information not available with batch methods. The risk assessment results of BSFL could help food legislation, relieve ecological concerns and food crises to some extent, and promote further research on other edible insects.

I090

QUANTIFICATION OF KEY ELEMENTS IN LITHIUM BRINES BY ICP-OES. Marc-Andre Gagnon, Neli Drvodelic, Longbo Yang. Agilent Technologies, 6705 Millcreek Drive, Mississauga, ON L5N 8B3, Canada. (longbo.yang@agilent.com)

Lithium is a key raw material in the production of high density, rechargeable batteries and battery packs. Currently lithium brine extraction is receiving increasing focus from the mining industry, due to its high efficiency and low environmental impact. Determining the elemental composition of the Li brine is crucial to the development and monitoring of the extraction process. However, the elemental testing for Li brine using ICP-OES technology is challenging due to its high total dissolved solid (TDS) content, high density and the likely presence of undissolved solids in the brine samples. Under such circumstances, using matrix-matching calibration standards containing high concentration of NaCl, is usually recommended. However, this approach is time-consuming and cannot efficiently account for the great variability of matrices encountered in real Li brine samples. The strategy presented here relies on the careful selection of an internal standard mixture to correct for physical interferences as well as other non-spectral interferences, e.g. the easily ionization element (EIE) effect, caused by large amount of NaCl in these samples. The solution proposed provides an excellent compromise between productivity, method detection limit, carryover management and sample preparation in order to deal with real-life Li brine samples under operational conditions.

SUCCESSFUL SCALE-UP AND DESTRUCTION OF PER- AND POLYFLUOROALKYL SUBSTANCES (PFAS) IN SOIL VIA BALL MILLING. Nicholas J. Battye¹, **Oliver J. Allshire¹**, David Patch¹, Dylan Roberts¹, Lauren Turner², Bernard H. Keuper², Kela P. Weber¹. ¹Environmental Sciences Group, Department of Chemistry and Chemical Engineering, Royal Military College of Canada, Kingston, ON K7K 7B4, Canada; ²Department of Civil Engineering, Queen's University, Kingston, Ontario K7L 3N6, Canada. (190ja1@queensu.ca)

Per- and polyfluoroalkyl substances (PFAS) are a class of fluorinated organic compounds used for their hydro-and-lipophobicity, low coefficient of friction, and surfactant abilities, among other properties. In recent years they have been classified as persistent organic pollutants due to their recalcitrance and toxicity to humans and the environment. Various remediation techniques have been investigated to remove or destroy PFAS in the environment, with a promising one being ball milling. Most PFAS ball milling has been conducted at a laboratory scale in controlled conditions, however for the technique to reach maturity it must demonstrate its effectiveness at an industrial scale with field soils. For this experiment, a horizontal ball mill at an industrial pilot scale of 240L was used. Two types of soil were used and the effect of KOH as a co-milling reagent was tested. High resolution mass spectrometry, liquid chromatography, and free fluoride analysis were used to inspect the degradation of a variety of PFAS compounds found in the soil, including PFOS, PFOA, 6:2 FTS, and 6:2 FTSAB. Results showed that moderate degradation of PFAS with KOH occurred at a rate of roughly 19 mg/kg of soil in one hour.

I092

⁸²Se-ENRICHED SELENIZED YEAST: A PERFECTLY MATRIX-MATCHED STANDARD FOR ISOTOPE DILUTION ANALYSIS OF SELENIZED YEAST. Kelly LeBlanc and Zoltan Mester. National Research Council Canada. (Kelly.LeBlanc@nrc-cnrc.gc.ca)

Dietary supplementation with selenized yeast has become increasingly common following epidemiological studies showing enhanced chemo-preventative effects of selenium when provided as a selenized yeast rather than in a pure chemical form. In typical commercially-available selenized yeasts, the majority (60-85%) of the selenium is present in the form of proteinaceous selenomethionine, which is notoriously difficult to analyze due to the requirement of breaking peptide bonds to release selenomethione in a way that is quantitative but also gentle enough to ensure no additional speciation changes occur. In analytical chemistry, isotope dilution analysis is commonly employed to ensure accurate results where analyte losses or changes can occur, but the use of a chemical spike is unable to account for incomplete recovery of selenomethionine from the proteinaceous form. Therefore, the National Research Council Canada has developed Certified Reference Material SEEY-1, which is a selenized yeast enriched with selenium-82. As a matrixmatched spike, SEEY-1 behaves in the same manner as a yeast sample during processing, meeting the isotope dilution requirement of sample/spike homogenization, accounting for potential incomplete recovery or analyte loss during sample preparation and allowing for more accurate quantitation of the selenium (total and individual species) in samples of selenized yeasts.

I093

OPTIMIZATION OF INFRARED-HEATED SAMPLE INTRODUCTION SYSTEM FOR INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY. **Darrian Prendergast**, Zichao Zhou, Diane Beauchemin Queen's University, Department of Chemistry, 90 Bader Lane, Kingston, ON K7L 3N6, Canada. (<u>18ddap@queensu.ca</u>)

This study demonstrates the effect of the structure of a modified cyclonic spray chamber on multielement sensitivity in inductively coupled plasma mass spectrometry (ICPMS.) Optimization of the infrared (IR)-heated sample introduction system can improve the transport efficiency of the technique by pre-evaporating the sample prior to its introduction into the plasma [1]. An IR-heated sample introduction system consisting of a ceramic bead IR-heater and an IR-heated modified cyclonic chamber was used for analysis of elements (Mg, V, Cr, Mn, Co, Ni, Cu, Zn, As, Se, Sr, Cd). Temperature optimization was conducted, and heating the sample introduction system to 60°C saw an improvement in the sensitivity of most elements by approximately 200% of that which was seen using the unheated system. The volume (50mL and 100mL) and distance between the baffle and the top surface of the spray chamber (2mm, 4mm and 6mm) were varied to determine their effect on multi-element sensitivity. The 50-6mm spray chamber showed the greatest improvements in sensitivity of the four.

[1] A. Al Hejami, M. J. Burgener, J. Burgener, D. Beauchemin, J. Anal. At. Spectrom., 35 (2020), 35, 1125

I094

DEVELOPMENT OF A METHOD FOR THE FORENSIC DISCRIMINATION OF SOLDER BY LASER INDUCED BREAKDOWN SPECTROSCOPY (LIBS). **Katie Moghadam**¹, Diane Beauchemin¹, and Claude Dalpé². ¹Queen's University, Department of Chemistry, Kingston, ON K7L 3N6, Canada; ²Royal Canadian Mounted Police, Ottawa, ON, Canada. (<u>14kkm3@queensu.ca</u>)

This research focuses on the development of an analytical method for the characterization and discrimination of solder. Solder is a valuable piece of evidence from a crime scene as it contains a suite of trace and bulk elements (e.g. Pb, Sn, Cu, Sb) that are signature to the material source. This creates an opportunity to quantify salvaged solders from different crime scenes or warrant searches, and as a result, associate samples that originate from a common source. LIBS will be explored as an alternative to past analytical applications used for solder analysis (optical emission and mass spectrometry) as it is non-destructive, avoids sample preparation, and requires minimal sample sizes. The instrument parameters and sampling method will be optimized to achieve high measurement precision and low detection limits, which will be demonstrated by the discrimination of solders using principal component analysis (PCA). PCA will further enable the identification of an "unknown" solder sample selected in a blind trial. This research offers a promising approach for the discrimination of solder in improvised explosive devices (IEDs), or in other salvaged evidence containing solder or similar metal alloys.

RISK ASSESMENT OF SEAWEED AS AN ALTERNATIVE PROTEIN SOURCE. **Helen Lord**, Qiqi Zhang, Andre Castillo, Kelly LeBlanc, Diane Beauchemin and Zoltan Mester. Queen's University, Department of Chemistry, 90 Bader Lane, Kingston, ON K7L 3N6, Canada; National Research Council of Canada, Ottawa, ON, Canada. (<u>17hgl1@queensu.ca</u>)

Elements such as Pb, Hg, Zn, Cd, As, Se, Ni and Cr have the potential to pollute alternative protein sources like seaweed. Artificial saliva, gastric and intestinal solutions are prepared and heated at 37°C. Using the on-line leaching method coupled to the inductively coupled plasma mass spectrometer (ICPMS), the maximum bio-accessible fraction of each analyte is measured in a seaweed sample. The ICPMS is equipped with a collision-reaction interface (CRI) with hydrogen as the reaction gas, minimizing carbon- and chlorine-based polyatomic interferences. The on-line continuous leaching method allows for real-time monitoring of potentially toxic elements as they leach out of the seaweed samples. Mass balance was verified at the 95% confidence level. Results obtained from seaweed showed the majority of elements were leached by saliva, followed by gastric juice. Minimal leaching occurred in intestinal juice. The on-line leaching method indicated that 18.9 - 97.4% of the analytes in seaweed are bio-accessible. However, comparison of bioaccessible fractions to tolerable uptake limits suggest that seaweed is safe to eat as a primary alternative protein source for all ages. Speciation analysis was performed on As and Se to determine what forms are leachable in seaweed during each saliva, gastric and intestinal phase. Inorganic and organic As species was monitored as well as SeMet. Comparisons of SeMet's proteinaceous form versus free SeMet was made in each gastro-intestinal phase.

I096

TOTAL CONSUMPTION INFRARED-HEATED SAMPLE INTRODUCTION SYSTEM TO IMPROVE INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY. **Zichao Zhou**, Ahmed Al Hejami and Diane Beauchemin, Queen's University, Department of Chemistry, 90 Bader Lane, Kingston, ON K7L 3N6, Canada (<u>16zz39@queensu.ca</u>)

Traditionally, a pneumatic nebulization system consisting of a pneumatic nebulizer and a spray chamber introduces sample solutions into inductively coupled plasma mass spectrometry (ICPMS). Unfortunately, 90-98% of the sample is wasted in the process, making the sample introduction system the Achilles' heel of ICP spectrometry. In this study, a total consumption infrared (IR) heated sample introduction system was optimized for ICPMS to increase sample transport efficiency while preserving plasma robustness by not removing water. A 50-mL cyclonic spray chamber was modified so that a pen IR heater could be introduced within the baffle in its centre. Optimized parameters included the sample uptake rate (25-75 μ L min⁻¹), IR heating temperature (20-300 °C), nebulizer gas flow rate (0.5-1.1 L min⁻¹), and sheathing gas flow rate (0-0.5 L min⁻¹). At 50 μ L/min, sensitivity for 22 elements (Li, Be, Sc, Ti, V, Cr, Mn, Fe, Ni, Ni, Co, Cu, Zn, Ga, As, Se, Y, Mo, Cd, In, Sb, Ce, Pt, Bi) doubled on average when the modified cyclonic spray chamber, sheathing device and base of the torch were IR-heated at 250°C. The effect of IR heating under total consumption conditions on detection limit, robustness, oxide formation and doubly-charge ion formation will be discussed.

DEVELOPMENT OF A SIMPLE METHOD FOR THE DIRECT DETERMINATION OF RARE EARTH ELEMENTS IN SLAG USING ELECTROTHERMAL VAPORIZATION INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETRY. **Yangyang Wang**, D. Beauchemin. Queen's University, Department of Chemistry, 90 Bader Lane, Kingston, ON, K7L 3N6, Canada. (<u>15yw103@queensu.ca</u>)

Inductively coupled plasma optical emission spectrometry (ICPOES) has been widely applied to rare earth element determination in refractory geological materials such as slag. The conventional sample introduction technique, pneumatic nebulization, is not compatible with slag samples, which are notoriously difficult to dissolve. Coupling the electrothermal vaporization (ETV) technique to ICPOES enables the direct analysis of solid slag samples. Without the lengthy acid digestion procedure, the ETV technique allows for faster analysis, reduces sample handling to eliminate chances of sample loss or contamination, and increases sample introduction efficiency, therefore, requiring less sample mass. Rare earth elements (Ce, Dy, Er, Eu, Gd, Ho, La, Lu, Nd, Pm, Pr, Sc, Sm, Tb, Tm, Y, Yb) in slag samples are determined based on 5-point external calibration curves constructed using certified reference materials (TILL 1, 2, 3 & 4). Point-by-point internal standardization is carried out with Ar 763.511 nm to correct for sample loading effect on the plasma. Using Minitab 19 statistical software, multivariate optimization was performed of the carrier gas, bypass gas, carbon tetrafluoride reaction gas flow rates, and pyrolysis temperature to maximize sensitivity. The developed method holds great promise for fast and easy determination of rare earth elements in slag and other refractory samples.

I098

COMPARISON OF THE MAXIMUM BIOACCESSIBILITY OF AS, CD, AND PB IN PEELED AND UNPEELED VERSIONS OF GARDEN PRODUCE FOUND IN YELLOWKNIFE, NORTHWEST TERRITORIES. **Andre Castillo**¹, Selena Domanski¹, Ashley Meness¹, Iris Koch², Mike Palmer³, Diane Beauchemin¹. ¹Queen's University, Department of Chemistry, 90 Bader Lane, Kingston, ON K7L 3N6, Canada; ²Environmental Sciences Group, Royal Military College of Canada, Kingston, ON, K7K 7B4, Canada; ³North Slave Research Centre, Aurora Research Institute, Aurora College, Yellowknife, NWT X1A 2R3, Canada. (<u>12aic1@queensu.ca</u>)

Elevated concentrations of As and other mine related contaminants found in regional soils in Yellowknife have resulted in the investigation of its long-term environmental impacts in the community. More specifically, its impact on the consumption of locally grown garden produce is of interest as it could pose a potential human health risk. The work presented will look to examine the maximum bioaccessibility of As, Cd, and Pb in garden produce grown in Yellowknife. This will be conducted using the continuous online leaching method (COLM) to determine maximum bioaccessibile concentrations and total concentrations of peeled and unpeeled version of carrots and potatoes. Bioaccessible concentrations and total concentrations of the garden produce had higher concentrations. In all cases, mass balance was verified to certify the results and the potential correlation of bioaccessibility in the peeled and unpeeled versions of garden produce will be examined.

A BIO-ACCESSIBILITY STUDY OF POTENTIALLY TOXIC ELEMENTS IN INSECT-BASED ALTERNATIVE PROTEINS USING INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY. **Cameron Pytyck**¹, Zoltan Mester², Diane Beauchemin¹. ¹Department of Chemistry, Queen's University, 90 Bader Lane, Kingston, ON K7L 3N6, Canada; ²National Research Council of Canada, Ottawa, ON, Canada. (<u>15crp5@queensu.ca</u>)

Bio-accessibility refers to the fraction that is dissolved in the gastro-intestinal tract and thus becomes available for absorption into the blood stream. A continuous on-line leaching method (COLM) was used to measure the bio-accessibility of elements present in multiple insect-based proteins. The method involved sequential leaching of black soldier fly larvae (BFLY-1), cricket powder (KRIK-1) and yellow mealworm (VORM-1) using different artificial gastro-intestinal fluids (like saliva, gastric juice, intestinal fluids) at 37°C to mimic the digestion process in the human body. Multi-element detection was done using an inductively coupled plasma mass spectrometry (ICPMS) instrument equipped with a dynamic-reaction cell (DRC) using oxygen as reaction gas to minimize polyatomic interferences. The COLM provides real-time monitoring of potentially toxic elements that are continuously released during leaching. The artificial gastro-intestinal reagents therefore were sequentially leached through a mini-column containing the sample and the leachates were directed into the ICPMS instrument for multi-element detection. The results showed that the bio-accessibility varies between each element and the greatest bio-accessible fractions were observed in the gastric phase. Residual and total digestions were also prepared and analyzed to verify mass balance. The sum of the bio-accessible fraction and remaining residual was in agreement with the total measured concentration for the majority of elemetns at the 95% confidence level for all samples. The percent recoveries were calculated and varied between each element, and the highest percentage was measured in the residual. Finally, the results obtained for the total digestions were compared to the certified values, and the majority were in agreement at the 95% confidence level for all samples.

I100

A METHOD FOR THE DETERMINATION OF ULTRA-TRACE RARE EARTH METALS IN REFRACTORY MATERIAL USING ETV-ICPOES. **Sophia Kienast**, Yanyang Wang, Diane Beauchemin. Queen's University, Department of Chemistry, 90 Bader Lane, Kingston, ON K7L 3N6, Canada. (<u>sophia.kienast@queensu.ca</u>)

Electrothermal vaporization coupled to inductively coupled plasma optical emission spectrometry (ETV-ICPOES) was used for the ultra-trace determination of rare earth metals (Ce, Dy, Er, Eu, Gd, Ho, La, Lu, Nd, Pm, Pr, Sc, Sm, Tb, Tm, Y, Yb) in slag. Slag is a by-product of mining; it contains the contents removed during the smelting process and is typically rich in metal oxides and sulphur. Using the ETV technique eliminates the need for sample preparation and greatly improves the efficiency of the measurements. Using Minitab 19 software, and after optimizing the reaction gas (carbon tetrafluoride), carrier gas, and bypass gas flow rates, as well as the pyrolysis temperature, we were able to reliably determine ultra-trace concentrations of the aforementioned rare earth metals in the slag sample. External calibration curves were generated using TILL 1, 2, 3, and 4 as reference materials.

ELUCIDATING DEGRADATION MECHANISMS FOR A RANGE OF PFAS VIA CONTROLLED IRRADIATION STUDIES. **David Patch¹**, Natalia O'Connor¹, Iris Koch¹, Tom Cresswell², Cath Hughes², Justin B. Davies², Jennifer Scott¹, Denis O'Carroll^{1,3}, Kela Weber^{1,3}. ¹Department of Chemistry and Chemical Engineering, Royal Military College of Canada, Kingston, ON, Canada K7K 7B4; ²Australian Nuclear Science and Technology Organisation, Australia; ³School of Civil and Environmental Engineering, University of New South Wales, Sydney NSW 2052, Australia. (David.Patch@rmc.ca)

Per- and polyfluoroalkyl substances (PFAS) are a challenging class of environmental pollutants due to a lack available destructive remediation technologies. Understanding the fundamental mechanisms for degradation of PFAS is key for the development of field scalable and in-situ destructive based remediation technologies. This study aimed to elucidate and refine the current understanding of PFAS degradation mechanisms in water through a series of controlled gamma irradiation studies. Gamma irradiation of PFAS was performed using a cobalt-60 source in a batch irradiation up to 80 kGy at the Australian Nuclear Science and Technology Organization. High resolution mass spectrometry was used to identify more than 80 fluorinated transformation products throughout the degradation experiments. Aqueous electrons were identified as the key reactive species responsible for initial PFAS degradation. Most importantly, based on degradation product formation, we found that the initial -F/+H does not have to occur at the α -fluoride (nearest the functional head group), rather occurring throughout the chain length leading to more complex degradation pathways than previously postulated. While our results support some of the reaction steps postulated in the literature, we have developed a unified 16 step and 3 pathway schematic of degradation supported by experimental observations.

I102

Parsimonious Methodology for Synthesis of Silver and Copper Functionalized Cellulose **David Patch¹**, Natalia O'Connor¹, Debora Meira², Jennifer Scott¹, Iris Koch¹, Kela Weber². ¹Environmental Sciences Group, Department of Chemistry and Chemical Engineering, Royal Military College of Canada, Kingston, ON K7K 7B4, Canada; ²Argonne National Laboratory, Lemont, IL 60439, USA. (David.Patch@rmc.ca)

Metal nanomaterials, such as silver and copper, are often incorporated into commercial textiles to take advantage of their antibacterial and antiviral properties. In this study, eight different methods were employed to synthesize silver, copper, and silver/copper functionalized cotton batting textiles. Using silver and copper nitrate as precursors, different reagents were used to initiate/catalyze the deposition of metal, including: (1) no additive, (2) sodium bicarbonate, (3) green tea, (4) sodium hydroxide, (5) ammonia, (6,7) sodium hydroxide/ammonia at a 1:2 and 1:4 ratio, and (8) sodium borohydride. The use of sodium bicarbonate as a reagent to reduce silver onto cotton has not been used previously in literature and was compared to established methods. For the silver treatment methods, bicarbonate and hydroxide resulted in the highest amounts of silver on the textile and for copper treatment the sodium hydroxide and sodium hydroxide/ammonium hydroxide resulted in the highest amounts of copper on the textile. Formation of copper oxide was dependent on the pH of the solution, with ammonia and other high pH solutions resulting in majority of the copper on the textile existing as copper oxide, with smaller amounts of ionic-bound copper.

CYCLIC VOLTAMETRIC AND ELECTROCHEMICAL IMPEDANCE SPECTROSCOPIC ANALYSIS OF ICE FORMATION ON SURFACE COATINGS TOWARD PREVENTION OF SAFETY HAZARDS IN THE AEROSPACE INDUSTRY. **Kate Yeadon**^{1,2}, Naiheng Song², Xiao Huang³, Edward P.C. Lai¹, Kailai Wang^{1, 1} Department of Chemistry, Carleton University, 1125 Colonel By Dr., Ottawa, ON, K1S 5B6; ² Aerospace Research Centre, National Research Council of Canada, 1200 Montreal Rd., Ottawa, ON, K1A 0R6; ³ Department of Mechanical and Aerospace Engineering, Carleton University, 1125 Colonel By Dr., Ottawa, ON K1S 5B6. (KATEYEADON@cmail.carleton.ca)

Ice accumulation on the surface is a source of safety hazards in many industries including aerospace, hydroelectric, transportation, and solar energy production. Passive technologies capable of reducing icing while reducing energy and lowering costs have gained interest. The most promising passive technology focuses on icephobic coatings to minimize the interaction between the water/ice-solid interface and to supress ice formation - or ice adhesion - on a surface. However, there is still a lack of understanding regarding icing behaviour during its initial stages. This research aims to determine changes to the exact temperature at which ice formation occurs on various surfaces - particularly icephobic coatings - using cyclic voltammetry and electrochemical impedance spectroscopy (EIS). The electron transfer behaviour of a redox probe in a water droplet on a surface can be measured by acquiring cyclic voltammograms. Initial analyses using this approach find that measured electrochemical behaviour on aluminum alloy substrates is altered following the application of commercially available icephobic coatings. A temperature-controlled icing set-up has been constructed in the lab to conduct similar experiments under icing conditions. By slowly lowering the temperature of the system, cyclic voltammograms and EIS analysis can be monitored continuously as the water droplet freezes to form ice. It is expected that the voltammograms and Nyquist plots will distort in shape during this phase change allowing us to pinpoint the exact temperature of ice formation. The comparison of these distortions across surfaces of different chemistry under different physical conditions can provide novel information about the initial stage of ice formation.

I104

CHALLENGES IN ATMOSPHERIC MERCURY SPECIATION ANALYSIS. Julia Lu. Department of Chemistry and Biology, Toronto Metropolitan University, Toronto, ON, Canada. (julialu@ryerson.ca)

Mercury (Hg) is a persistent, toxic and bioaccumulative pollutant. It exists in several physical states and chemical forms depending on the environmental conditions. The atmosphere is the major pathway for its transport from sources of emission to the various environments. The three most common forms of mercury in the atmosphere are gaseous elemental mercury (GEM, Hg0), gaseous oxidized mercury (GOM) or reactive gaseous mercury (RGM), and particle-bound mercury (Hgp, TPM). In this presentation, measurement techniques for GEM, GOM and TPM will be briefly reviewed and challenges for identifying and quantifying of these species be discussed.

I105

EXPANDING THE USABILITY OF SEDIMENT CERTIRIED REFERENCE MATERIALS WITH SEQUENTIAL EXTRACTION DATA. **Kelly LeBlanc**, Indu Pihillagawa Gedara, Ovi Mihai, Paramee Kumkrong, and Zoltan Mester. National Research Council Canada. (Kelly.LeBlanc@nrc-cnrc.gc.ca)

Sequential extraction experiments have the ability to provide an abundance of environmentallyand geologically-relevant information about sediments and soils. These multi-step procedures allow researchers to distinguish between metals (and other contaminants) which are easily leachable or strongly bound to sediments, and which are associated with different physiochemical compartments of these materials. The three step BCR (developed by the European Community Bureau of Reference [1]) and four step Tessier (from Tessier *et al.* [2]) procedures are among the most commonly employed sequential extraction protocols used for trace metals in sediments and soils. Unfortunately, a lack of Certified Reference Materials (CRMs) available with information relating to leachability has meant laboratories implementing these protocols have been lacking appropriate quality control samples for method validation. Therefore, the National Research Council Canada has recently updated the certificates for sediment CRMs HISS-1, MESS-4, and PACS-3 with data obtained following both BCR and Tessier sequential extraction protocols. This talk will discuss the road that lead to the update of our certificates, the challenges associated with this type of analysis, and the importance of community data in the maintenance of CRMs.

[1] European Comission. 2001. BCR-701.

[2] Tessier et al. 1979. Anal. Chem. doi 10.1021/ac50043a017

I106

ARSENOBETAINE: SYNTHESIS AND SELECTIVITY. **Blaire Coffey**, Jennifer Scott, and Iris Koch. Royal Military College of Canada, Department of Chemistry and Chemical Engineering, Environmental Sciences Group, PO Box 17000, Station Forces, Kingston, ON K7K 7B4, Canada. (blaire.coffey@rmc.ca)

Arsenic is a widely distributed element within the environment. This metalloid can be found in a variety of chemical forms. Arsenic is commonly known as a poison and carcinogen; however, arsenic compounds display different toxicities. Generally, inorganic arsenic forms, including arsenite and arsenate, are more toxic. Organoarsenic compounds, specifically those in the pentavalent form, are less toxic. One organoarsenic compound, arsenobetaine (AB), exhibits nontoxic properties. AB is predominantly found in marine organisms and less commonly found in the terrestrial environment with the exception of fruiting bodies (or mushrooms) of some terrestrial fungi species. Currently, AB's role and formation in mushrooms is unknown. It is hypothesized that AB acts as an osmolyte within the mushroom. This presentation examines potential abiotic formation pathways for AB. One proposed pathway involves exposing trimethylarsine oxide to a haloacetic acid in the presence of glutathione. To investigate this pathway, various synthetic reactions were conducted. These reactions were analyzed for arsenic speciation using HPLC-ICP-MS targeting AB. Additionally, a field portable x-ray fluorescence based method was developed to provide real-time analysis of AB in solid and liquid samples. This method was verified using synchrotron-based x-ray absorption near edge structure analysis at the Advanced Photon Source in Chicago, IL.

DETERMINATION OF CHROMIUM SPECIES IN DRINKING WATER BY ION CHROMATOGRAPHY (IC)–ICP-MS (FAST, SENSITIVE, AND ACCURATE MEASUREMENT OF CR(VI) USING A METROHM IC COUPLED TO AGILENT ICP-MS). **Parviz Shahbazikhah**¹, Jayesh Gandhi², Yan Cheung³. ¹Metrohm Canada; ²Metrohm USA, Riverview, Florida, USA; ³Agilent Technologies, USA. (parviz.shahbazikhah@metrohmca.com)

Chromium (Cr) is a metallic element that occurs naturally in rocks, soils, plants, and man-made salts. There are two main stable forms or species of Cr: trivalent chromium (Cr(III)) and hexavalent chromium (Cr(VI)). Cr(III) is found in soil, water, vegetables, fruits, and meats, and is essential to human health as it helps to convert fat into energy. However, Cr(VI) form is toxic and known carcinogen and needs to be monitored at lowest detection level as possible. There is an USEPA method for measuring Cr(VI) by IC UV- PCR technology, however, IC-UVPCR technology does not measure Cr(III) specie. ICP-MS is routinely used for the measurement of Total Cr at low concentration levels. ICP-MS can also be coupled to a chromatographic separation technique such as high performance liquid chromatography (HPLC) to provide specific detection and quantification of Cr(III) and Cr(VI). In this study, ion chromatography (IC) was coupled to ICP-MS for separation and low-level detection of Cr(III) and Cr(VI). Compared to HPLC, IC has some advantages for routine Cr speciation, as the IC pump is relatively low cost, and the IC sample path is metal-free. The metal (e.g. stainless steel) parts of a standard HPLC system can be replaced with metal-free components, but this adds further cost. A metal-free IC system coupled to an ICP-MS provides a cost-effective solution for routine, low-level analysis of the individual Cr species. In this presentation, we will present Method development, the method detection limits (MDLs) and data from "Real World Samples" that could be achieved for Cr(III) and Cr(VI) by IC-ICP-MS.

I108

HOT TOPICS IN HEAVY METALS TESTING IN FOOD. Jenny Nelson¹, Elaine Hasty², Macy Harris², Sam Heckle², and Leanne Anderson². ¹Agilent Technologies Inc.; ²CEM Corporation, USA. (jenny.nelson@agilent.com)

We will describe the use of the Agilent 7850 ICP-MS for the analysis of metals in a variety of hot applications in food. Alternative Proteins: There are rising numbers of people consuming vegan or vegetarian diets, or who are reducing their intake of animal-based foods. This food trend can be seen in the ever-increasing selection of alternative protein foods in grocery stores, on the menus of fast-food outlets, and in many restaurants. **Baby Food**: South of the border in the US, the government is expected to introduce stringent action limits for iAs, Cd, Hg, and Pb in baby and infant foods. Therefore, manufacturers of baby food products urgently require easy-to-use, reliable, and accurate methods for the routine testing of these metals and compounds in ingredients and final products. **Edibles**: There is an absence of regulatory methods for the accurate testing of Cannabis products, for many toxins, like heavy metals. AOAC and ASTM have taken on this task and have been diligently working towards this goal. We will present our methods for the preparation and multi-elemental analysis of edible products prepared for analysis using microwave-assisted acid digestion followed by testing with ICP-MS.

I109

10 YEARS OF ICP-MS/MS: HOW AGILENT REVOLUTIONIZED ICPMS. **R. Bastian Georg**, Clint Walker, Ed McCurdy, Glenn Woods and Jean-Louis Cabral. Agilent Technologies, Canada, 6705 Millcreek Dr., Mississauga, ON L5N 5M4, Canada (<u>Bastian.Georg@Agilent.com</u>)

In 2012, Agilent forever changed ICPMS with the introduction of the first ever commercial ICP-MS/MS platform, the Agilent 8800. From the patented 5-stage vacuum system to the unique tandem configuration consisting of two full-sized quadrupoles, the Agilent 8800 was built to overcome limitations of common ICPMS platforms. The Agilent 8800 instrument combined the ease-of-use and scan speeds of quadrupole based ICPMS with interference handling capabilities outperforming high resolution sector-field ICPMS systems, providing unmatched ICPMS performance. The ICP-MS/MS technology was quickly adopted into laboratories across the globe, resulting in a strongly growing number of research publications and newly emerging ICPMS applications. Taking the lessons learned from the 1st ever ICP-MS/MS, Agilent introduced a new, more powerful, generation of ICP-MS/MS systems, the Agilent 8900, in 2016. Join us for a 10th anniversary celebration of ground-breaking ICP-MS/MS technology and learn how Agilent's ICP-MS/MS pushes ICPMS analyses to new heights.

Happy 10th Birthday ICP-MS/MS!

I110

THREE-DIMENSIONAL NANOSCALE MORPHOLOGY CHARACTERIZATION OF TERNARY ORGANIC SOLAR CELLS. **Ting Yu**, Wanting He, Ricardo Izquierdo, Dongling Ma. Institut national de la recherche scientifique (INRS-EMT), 1650 Bd Lionel-Boulet, Varennes, QC J3X 1P7. (ting.yu@inrs.ca)

Developing ternary organic solar cells (OSCs) is a promising strategy to improve the photovoltaic performance. The advanced characterization of photoactive layers allows a better understanding of morphology, and thereby more effective realization of high-performance solar cells. In this work, we integrated perylenediimide (PDI)-based non-fullerene material into OSCs as the third component to form the ternary OSCs. [1] Specifically, three-dimensional nanoscale morphology of ternary film was characterized in depth by photo-induced force microscopy (PiFM) coupled with infrared laser spectroscopy and the energy-filtered transmission electron microscopy (EFTEM), which qualitatively and quantitatively "view" the surface and cross-sectional morphology, and provide strong evidence that PDI-based material can suppress the aggregation of the fullerene molecules and generate the homogenous morphology with a higher-level of the molecularly mixed phase. In particular, the application of PiFM in this study enables the chemical imaging of all the three components at a high spatial resolution (~10 nm), unprecedented by all previous chemical imaging techniques. Thus, our work represents the first exploration of the morphology characterization in the real device setting with advanced characterization techniques.

[1] T. Yu, W. He, M. Jafari, M.Siaj, R. Izquierdo, B. Sun, G. C. Welch, A. Yurtsever, D. Ma, *Small Methods* 6 (2021), 2100916.

I111

FERROFLUID BASED PRECONCENTRATION AND ULTRA-TRACE DETERMINATION OF POTENTIALLY TOXIC INORGANIC ARSENIC, CHROMIUM, AND SELENIUM IN ENVIRONMENTAL WATERS BY INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY, **Yam Gotame** and Diane Beauchemin, Queen's University, Department of Chemistry, 90 Bader Lane, Kingston, ON, K7L 3N6, Canada. (15yng@queensu.ca)

This research focuses on developing a method for the ultra-trace determination of potentially toxic inorganic As, Cr, and Se in environmental samples through selective preconcentration using a ferrofluid. Specifically, Fe_3O_4 magnetic nanoparticles (MNPs) coated with silica and functionalized with L-cysteine are employed for the selective sorption of target analytes. After injection of the ferrofluid into an aqueous sample, the functionalized MNPs adsorbed the target analytes within a few minutes. It is found that in pH range 2-6, only As(V), Cr(VI), and Se(VI) selectively adsorbed on MNPs and eluted using 10% (v/v) HNO₃ and then separated using an external magnet. The unretained As(III), Cr(III), and Se(IV) are oxidized using 1M KMnO₄ to get the total concentration of As(VI), Cr(VI), and Se(VI). The resulting eluate was analyzed by inductively coupled plasma mass spectrometry. Preliminary results indicated a more than 70% recovery for all analytes. The results obtained after optimization of the extraction temperature, pH, eluent composition, elution time, and elution volume will be presented.

I112

AIR QUALITY MONITORING, SCIENCE, AND OUTREACH IN WATERLOO REGION, ONTARIO. **Yara Khalaf**¹, Carol Salama¹, Brenda Kurorwaho¹, Jessica D'eon², and Hind A. Al-Abadleh¹. ¹Department of Chemistry and Biochemistry, Wilfrid Laurier University, Waterloo, ON N2L 3C5; ²Department of Chemistry, University of Toronto, Toronto, ON M5S 3H6. (khal6090@mylaurier.ca)

Waterloo Region in Ontario is ranked third in Canada in terms of population growth, yet it only has one provincial air quality monitoring station. In my presentation, I will describe a pilot project launched in September 2020, when our group installed a network of AQMesh Multisensor Mini Monitoring Stations (pods) near elementary schools in Kitchener, the largest city in Waterloo Region. The major research findings suggested the need for additional air quality monitoring infrastructure in hot spots and for addressing analytical chemistry challenges in sensor performance [1]. As a result, an outreach and educational project (The Clean Air Project) started in May 2021, which targets elementary students attending schools near the installed pods [2]. The overarching goals of the outreach project were to increase scientific understanding, spread awareness about air quality, and promote long-term behavioural changes related to environmental sustainability. The project provided several benefits that include encouraging a healthy environment, fostering practical lessons in classrooms, and creating an environment that reduces the transmission rate of illnesses. The feedback from the students and the teachers was used to inform city and regional officials about possible policy changes to improve air quality in their communities.

[1] Mohammed, W.; Shantz, N.; Neil, L.; Townend, T.; Adamescu, A.; Al-Abadleh, H. A. *Atmosphere*. **2022**, *13*, 83; <u>https://doi.org/10.3390/atmos13010083</u>.

[2] Hind A. Al-Abadleh*, <u>Yara Khalaf, Carol Salama, and Brenda Kurorwaho</u>. *Science*, **2022**, 376(6593), 589.

PRECONCENTRATION OF NOBLE METALS ANALYZED BY INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY WITH FLOW INJECTION ANALYSIS. **Madison Langley**, Michael Trolio, Diane Beauchemin. Queen's University, Department of Chemistry, 90 Bader Lane, Kingston ON K71 3N6, Canada. (madison.langley@queensu.ca)

Analytical boundaries are present in the determination of noble metals in environmental samples such as their occurrence at low concentrations and complex matrices. With the increasingly high demand for these metals, their low abundance, and no suitable substitutes analytical techniques must be developed to overcome these boundaries. Inductively coupled plasma mass spectrometry (ICP-MS) was coupled with flow injection analysis (FIA) to optimize a preconcentration method for the analysis of noble metals Ag, Au, Ir, Os, Pd, Pt, Re, Rh, and Ru. The samples were preconcentrated through the formation of chloro-complexes using 0.01 M HCl, loaded onto an alumina column, eluted with a mixture of thiourea and aqua regia and then analysed using coupled ICP-MS and FIA. Through the use of alumina as a sorbent and a mixture of thiourea and aqua regia as eluents, recovery of all the noble metals were possible and 3-25 times precondition was observed which can be used for the accurate determination in geological samples. Validity of the method was confirmed through the analysis of CDN-PGMS-19 ore reference material.

I114

ASSESSING OUR EXPOSURE TO ENVIRONMENTAL CONTAMINANTS IN URINE BY TARGETED AND UNTARGETED LC-MS/MS METHODS. Lekha Sleno, Ons Ousji, Ikram Benhadji Serradj, Department of Chemistry, Université du Québec à Montréal, Montréal, QC, Canada. (sleno.lekha@uqam.ca)

We are continuously exposed to a myriad of environmental contaminants, which can pose serious health risks. The focus of this study was to develop workflows to assess our exposure to several environmental contaminants. Urine samples from volunteers were collected to monitor exposure to several classes of contaminants (including plasticizers, drugs, PCPs, and pesticides) with simple protein precipitation sample preparation. Targeted LC-MRM was used to assess the presence of parent compounds before and after enzymatic hydrolysis, using two different mobile phase systems for the best coverage. Untargeted analyses were also performed on a quadrupole-time-of-flight platform to detect contaminants and metabolites. Specific examples of contaminants include butylated hydroxytoluene (BHT), triclosan and oxybenzone, commonly used in PCPs. For several targeted compounds, detection of their metabolites yielded higher sensitivity than the non-metabolized parent compounds. A highly multiplexed targeted analysis is being developed for contaminants and their main metabolites in urine.

PHOTOCHEMICAL VAPOR GENERATION FOR ELEMENTAL ANALYSIS: THE ROAD SO FAR... Daniel L. G. Borges, Universidade Federal de Santa Catarina, CFM - Chemistry Department, Florianópolis-SC, Brazil. (daniel.borges@ufsc.br)

Sample introduction remains as a critical step in analytical methods associated to inductively coupled plasma mass spectrometry (ICP-MS) and the search for a straightforward technique that would allow analyte/matrix separation prior to ICP-MS detection has led to the development of photochemical vapor generation (PVG) systems that could be coupled to ICP-MS. Photochemical vapor generation relies on the formation of volatile species of ionic analytes solubilized in solutions containing (typically) low molar mass carboxylic acids upon UV irradiation. Our group has focused on the investigation of PVG using a simple photoreactor assembled using two 40 W UV lamps emitting at 257 nm, on top of which a quartz capillary with a capacity of 6.5 mL is positioned. Acetic and formic acids, either individually or mixed in concentrations that range up to 40% have been evaluated as to its efficiency in forming volatile species of elements such as Ni, Co, Fe, Hg and Te and also Au, Pd, Pt, Rh and Ir. The former group of elements benefited from a mixture of acetic and formic acid at 15% v/v each, whereas the noble metals were photochemically derivatized using formic acid individually. Flow through lamps, which offer the possibility of higher irradiation efficiency and a 185 nm photon beam, were also investigated. In this case, bromide and bromate could both be transferred (as a bromine compound) to the gas phase efficiently. Metallic sensitizers, such as Pt²⁺ or Cu²⁺, were investigated and provided increased overall photochemical efficiency for the analytes, likely due to the formation of bromo-complexes and charge/atom transfer processes. Osmium has also been photochemically volatilized upon reduction conditions, resulting in a species that remains to be identified. Up to now, one of the main goals of PVG is to produce a robust method, capable of surpassing effects such as reaction suppression by concomitant species, particularly oxygenated anions. The results obtained for a varied set of samples, including oily sludge leachates, seawater, biodiesel, digested biological tissues and alcoholic beverages, will be discussed. Overall, PVG was proven effective for the formation of volatile species of the investigated analytes and it is, therefore, an interesting alternative to provide analyte-matrix separation.

I116

LOOKING INSIDE CANADIAN OILSEEDS – OIL PROFILING BY HRMAS NMR. **Christopher W. Kirby**,* Leah K. Gauthier, and Maike B. Fischer, Charlottetown Research and Development Centre, Agriculture and Agri-Food Canada, 440 University Avenue, Charlottetown, PEI, Canada C1A 4N6 (Chris.Kirby@agr.gc.ca)

The fatty acid profiles of intact small Canadian oilseeds (flax, canola, camelina, and borage) have been determined using ¹H and ¹³C HRMAS NMR methods. The experiments were conducted on a 4 mm H/C/P HRMAS probe without the use of HRMAS inserts. The solid-state NMR rotors used contained a single oilseed as well as multiple seeds (number of seeds depending on seed size). Fatty acid profiles of the single oilseeds vs multiple oilseeds compare well. After the oilseeds were spun in the NMR magic angle spinning probe @6000 Hz, a germination test was performed to show that the NMR tested seeds had the same viability as untested oilseeds. These results will be compared to the usual solution ¹H NMR method following oil extraction from oilseeds to show that one does not need to destroy the oilseed to determine fatty acid profile within the seed.

ACCELERATED SIZE-FOCUSING PHOTOCHEMICAL SYNTHESIS OF NOVEL FLUORESCENT PEPTIDE-PROTECTED GOLD NANOCLUSTERS FOR BIOMEDICAL APPLICATIONS. **Parimah Aminfar**, Kevin Stamplecoskie. Department of Chemistry, Queen's University, Kingston, ON K7L 3N6, Canada. (18pa17@queensu.ca)

The synthesis and isolation of atomically precise gold and silver metal clusters have recently attracted enormous research interest. This is especially true for aqueous soluble clusters that typically display more challenges in achieving pure, atomically precise samples and in crystallization for characterization. Owing to their tunable optical, electronic, and physiochemical properties thiolate-protected gold clusters have been highly attractive for biomedical applications compared to traditional gold nanoparticles. Before clinical use, it is important to ascertain that these clusters are pure and single size to be able to track them in cells and understand their function in the body. In this work, we propose light activated synthesis of Au clusters followed by an accelerated size-focusing step using a sequence of peptides as a ligand. Using Norrish type I photochemistry, a unique control over the most challenging aspects of metal cluster synthesis is provided. The obtained Clusters were found to be optically pure using fluorescence excitation-emission matrix (EEM) spectroscopy and PARAFAC analysis. These novel clusters were then characterized by high resolution Electrospray Ionization (ESI) and Time-of-Flight (TOF) Mass Spectrometry and were prepared to be tested in a cell environment.

I118

SPIN-POLARIZED EMISSION IN NOBLE METAL NANOCLUSTERS. Patrick J. Herbert and **Kenneth L. Knappenberger, Jr.** Pennsylvania State University, Department of Chemistry, University Park, PA 16802, U.S.A. (klk260@psu.edu)

Spin-state-resolved electron dynamics and optical properties of metals confined to the naometerlength scale will be described. Sub-nanometer metals exhibit electronic, magnetic, and optical properties that are determined by atomic-level structure. Electronically excited metal clusters relax through a series of competitive internal conversion and intersystem crossing pathways, resulting in varying degrees of transient spin polarization. In particular, spin polarized emission for these systems can be modulated by metal-atom substitution and ligand exchange.[1,2] The ability to control electron dynamics in nanoclusters is expected to have impacts for metal-mediated catalysis, quantum information technologies, and many other applications. Future directions toward controlling macroscopic properties through microscopic structure will be presented.

[1] P. J. Herbert, K.L. Knappenberger, J. Phys. Chem. C. 125 (2021) 7267-7275.
[2] P.J. Herbert, K.L. Knappenberger Small, (2021) 2004431.

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I119 P

FORENSIC ANALYSIS FOR SEX IDENTIFICATION OF CHILD HAIR USING ELECTROTHERMAL VAPORIZATION COUPLED TO INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETRY (ETV-ICP-OES). Emilie Brossier, Yangyang Wang, Diane Beauchemin, Queen's University Department of Chemistry, 90 Bader Lane, Kingston, ON K7L 3N6, Canada. (diane.beauchemin@queensu.ca)

Previous research has allowed for the development of methods which enable the identification of an adult individual's sex based on a small hair sample. One of these methods uses electrothermal vaporization coupled to inductively coupled plasma optional emission spectrometry (ETV-ICPOES) which analyzes the trace elements in the hair samples. The peak areas of the trace elements can be used to determine which ones are sex-dependent, therefore enabling the prediction of the sample's sex without the presence of any deoxyribonucleic acid (DNA). In this study the method developed previously for adult hair was tested on child hair samples. Adult hair samples were first analyzed using linear discriminant analysis (LDA) and principal component analysis (PCA) to determine which elements could be used to accurately predict the sex. The method was then applied to child hair.

I120

NEW INSIGHTS INTO ESTIMATING THE TIME SINCE DEPOSITION OF SPECTROSCOPY BLOODSTAINED **EVIDENCE USING** APPLIED AND ELECTROCHEMISTRY. Theresa Stotesbury. Ontario Tech University, Faculty of Science -Forensic Science Program, 2000 Simcoe Street North, Oshawa, ON, L1G 0C5, Canada. (theresa.stotesbury@ontariotechu.ca)

The identification and quantification of (bio)molecules and their metabolism, formation, degradation and persistence are important features of forensic evidence; and in particular, are useful in time series estimation approaches to date evidence. For example, estimating the time since deposition (TSD) of a bloodstain can provide important medicolegal information for crime scene investigation (e.g. answering the question, "When did the bloodshed occur?"). Research in this area primarily investigates either hemoglobin OR genetic material degradation over time. In this talk I will present overviews of two recent studies from our laboratory that contribute to the TSD literature. The first will focus on combining hemoglobin (alpha and beta bands) and DNA degradation (base pair size) metrics using UV-VIS spectroscopy and high-resolution automated electrophoresis to improve the accuracy of linear mixed TSD models. The second will focus on using a novel electrochemical method using differential pulse voltammetry for TSD modelling with a clear diagnostic change at approximately 96 hours. In all cases, adding the random effect of the biological replicate accounted for a large proportion of the variation in signal. Effects of environmental and storage conditions relevant to crime scene investigation will also be discussed.

PHOTOLUMINESCENCE ENHANCEMENT AND PRESERVATION IN MONOLAYER MOLYBDENUM DISULFIDE VIA SUPERACID TREATMENT AND 2D ENCAPSULATION. Kurt H. Tyson, James R. Godfrey, Robert. G. Knobel, and **James M. Fraser**. Queen's University, Department of Physics, Engineering Physics & Astronomy, 64 Bader Ln, Kingston, Ontario, Canada, K7L 3N6. (james.fraser@queensu.ca)

Atomically thin 2D materials often display unique and exotic properties due to quantum confinement effects not present in their bulk counterparts. Monolayer transition metal dichalcogenides (TMDCs) such as MoS_2 have received significant attention as a layer-dependent direct band gap semiconductor as they are the natural limit for ultrathin and flexible optoelectronic devices [1] while also benefitting from low-cost/complexity fabrication (e.g., exfoliation). However, due to their single atomic thickness, TMDCs suffer from inherently large defect densities which limits their photoluminescence (PL) quantum yield to <1% [2]. This problem diminishes their ability to emit and absorb light necessary for potential next-gen photodetectors, emitters, lasers, etc. Using a bis-trifluromethanesulfonimide superacid treatment, we demonstrate more than an order of magnitude increase in photoluminescence. Additionally, we partially preserve the PL enhancement with hexagonal boron nitride (hBN) encapsulation. The origin of this enhancement is clarified through both steady-state and time-resolved spectroscopy.

[1] E. Singh, P. Singh, K. Kim, G. Y. Yeom, and H. S. Nalwa. *ACS Appl. Mater. Interfaces* **11** (2019), 11061–11105.

[2] M. Amani et al. Science **350** (2015), 1065-1068.

I122

A NEW DIMENSION IN HETEROGENEOUS CATALYSIS. **Frédéric A. Perras**, Alexander L. Paterson, James Cunningham, Guillaume P. Laurent, Scott Southern, Takeshi Kobayashi, Marek Pruski, Aaron D. Sadow, Igor I. Slowing, US DOE, Ames Laboratory, Ames, IA 50011 (fperras@ameslab.gov)

Crystallography has enabled unparalleled precision in the development of structure-activity and selectivity relationships for homogeneous catalysts and the design of bespoke ligands for stereoselective catalysis. Meanwhile, heterogeneous catalysis has seen so such revolution. Our understanding of the structures of single-site heterogeneous catalysts is largely based on the structures of the precursor complexes, their behavior, and imprecise spectroscopic methods. With the development of the highly sensitive dynamic nuclear polarization (DNP) surface-enhanced NMR spectroscopy (SENS) method, the determination of high-resolution, three-dimensional, structures from surfaces sites is becoming possible. This presentation will go over some of the recent developments from our lab aimed at the measurement of precise internuclear, and surface-to-atom distances for the goal of the determination of crystallography-like structures of supported metal complexes. These methods enable for the determination of configurations, conformations, orientations, and coordination geometries from these metal complexes and provide valuable structural details for computational studies of reaction mechanisms. We will conclude by showing a recent example where the determination of the three-dimensional structure of a supported enantioselective catalyst was able to explain its enhanced selectivity when grafted onto silica.

A COMBINED THEORETICAL AND EXPERIMENTAL CHARACTERIZATION METAL-WITH FRAMEWORKS POTENTIAL **APPLICATION** ORGANIC (MOFS) TO SUPERCAPACITORS. Carlos M. Hangarter¹, Boris Dyatkin¹, Anna Albert¹, Matthew Laskoski¹, Mark C. Palenik¹, Joel B. Miller¹, Michael W. Swift², John L. Lyons², and Christopher A. Klug¹. ¹Chemistry Division, U.S. Naval Research Laboratory, Washington, DC, USA; ²Materials Science Division. U.S. Naval Research Laboratory, Washington, USA DC. (christopher.klug@nrl.navy.mil)

Among the many exciting potential applications for metal-organic frameworks (MOFs) is their use in electrodes for energy storage. I will present results from the synthesis and characterization of the three different MOFs, 1) a Zr-MOF containing Zr₆O₄(OH)₄ clusters with 2,2'-bipyridine-5,5'-dicarboxylate linkers, 2) a Co-LMOF containing Co(H₂O)₂/H₂O clusters with hexamethylenetetramine and 2,3,5,6-tetrafluoroterephthalic acid linkers and 3) a simpler zeolitic imidazolate framework, ZIF, with Co²⁺ metal nodes and 2-methylimidazolate linkers. Gas sorption measurements, powder X-ray diffraction (XRD), scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS), and cyclic voltammetry were used to characterize our materials and results are compared to those in the literature. In addition, we used solid-state nuclear magnetic resonance (NMR) to determine the local structure and dynamics of these materials. Comparisons were made between the observed ¹H and ¹³C NMR chemical shifts and density function theory (DFT) calculations. Ultimately, while we found that the electrochemical performance of the materials varied considerably with the ZIF being the most promising. We confirmed the applicability of solid-state NMR to characterize the local structure and dynamics of these and similar materials as we begin full in situ NMR studies of MOF-based electrodes.

I124

GOING *AGAINST* THE FLOW: A RETURN TO A MEMBRANE APPROACH FOR MICROFLUIDIC MICROBIAL FUEL CELLS YIELDS NEW BENCHMARKS. L. Gong, M. A. Amirdehi, J. M. Sonawane, N. Jia, L. Torres de Oliveira, **J. Greener.** Département chimie, Université Laval, Pavillon Alexandre-Vachon 1045, avenue de la médecine, local 4064^E, Québec, G1V 0A6, Québec. (jesse.greener@chm.ulaval.ca)

An ongoing trend in microfluidic microbial fuel cells (MFCs) is to exclude the separation membrane between anode and cathode compartments, and instead rely on the physics of laminar flow to maintain isolation between anolyte and catholyte streams. Nevertheless, diffusion and inadvertent flow disturbances can result in mass transport crossover. To avoid this, membraneless electrodes in MFCs are regularly separated by distances of several millimeters, but this negatively affects the internal resistance and undermines a prime advantage of microscale MFCs. Here I will demonstrate a new idea that uses an *in situ* synthesised biocompatible micromembrane supports submillimeter electrode spacing. The sub-millimeter membrane MFC under comparable flow conditions to the best performing membraneless microfluidic MFC had an internal resistance that was 60% lower, power and current densities that were respectively 45% and 290% higher, and acetate conversion efficiencies that were 8 times higher. The resulting flow stability enabled continuous increases to power density of up to 30% for flow rates of 100 times over the baseline. As a result, maximum outputs obtained were 660 mW m⁻¹ and 3.5 A m⁻¹, the highest reported values for microfluidic MFCs using pure culture bacteria.

ASSESSING THE PROPERTIES OF NANOCLUSTERS FOR PHOTODYNAMIC THERAPY **Kristen Harrington** and Kevin Stamplecoskie. Queen's University, Department of Chemistry, Kingston, ON, Canada. (16kvh1@queensu.ca)

Every year more than 200,000 Canadians are diagnosed with cancer. Despite many advances in cancer research, there is still need for improved cancer treatment options. This project involves the synthesis and characterization of metal nanoclusters, which have been proposed as photodynamic therapy (PDT) agents, which is a type of cancer therapy. As clusters are being explored for their use in PDT, it important to identify clusters that are both stable in physiological conditions and able to produce reactive oxygen species, the active compounds in PDT. Here, we use absorbance and emission spectroscopy to monitor the stability of clusters in conditions that mimic physiological conditions. We also quantify the production of reactive oxygen species from different clusters to determine if they will be effective PDT agents. Overall, cluster stability and reactive oxygen species production varies with cluster structure and ligand. This work provides useful information in selecting nanoclusters that will be suitable for PDT.

I126

HIGH FIELDS, FAST SPINNING AND PULSE SEQUENCES FOR ENHANCING NMR SENSITIVITY AND RESOLUTION OF QUADRUPOLAR NUCLEI. **Zhehong Gan**, National High Magnetic Field Laboratory, Tallahassee, Florida, USA (gan@magnet.fsu.edu)

High magnetic fields can drastically enhance NMR spectral resolution and sensitivity particularly for quadrupolar nuclei. The National High Magnetic Field Laboratory (NHMFL) has developed and commissioned a 36 T series-connected hybrid (SCH) magnet for NMR applications. A collection of solid-state NMR of insensitive quadrupole nuclei such as ¹⁷O, ⁶⁷Zn, ⁴³Ca and their applications to metal-organic-frameworks (MOFs), catalysts, and bio-solids will be presented to demonstrate the advantages of resolution and sensitivity by the 1.5 GHz high field [1]. Solid-state NMR methods and pulse sequences to improve the efficiency and resolution of insensitive quadrupolar nuclei will also be presented, including robust TRAPDOR-HMQC and multi-nuclei correlation via ¹H detection under fast magic-angle spinning [2] and the use of low-power and long pulses for efficient multiple-quantum magic-angle spinning (MQMAS) [3].

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OXYGEN-17 NMR STUDIES OF PROTEINS: OPPORTUNITIES AND CHALLENGES. Gang Wu, Department of Chemistry, Queen's University, Kingston, ON K7L 3N6, Canada (wugang@queensu.ca)

We have recently demonstrated that it is feasible to achieve amino acid-type specific ¹⁷O-labeling of proteins via recombinant expression in an auxotrophic *Escherichia coli* strain. This new approach allows incorporation of ¹⁷O isotopes into both the protein backbone and side chains. This opens up new opportunities for ¹⁷O NMR studies of proteins. In this talk, we will discuss this new ¹⁷O-labeling approach and present ¹⁷O NMR results obtained at high magnetic fields up to 35.2 T.

I128

NANODISCS BASED BIOLOGICAL NMR STUDIES. (Rams) Ayyalusamy Ramamoorthy, Biophysics, Department of Chemistry, Biomedical Engineering, Macromolecular Science & Engineering, and Michigan Neuroscience Institute, University of Michigan, Ann Arbor, MI 48109 (ramamoor@umich.edu)

Despite recent developments in structural biology, probing dynamic protein-protein and proteinmembrane interactions that play vital roles on the pathomechanisms of various diseases has been a major challenge to most biophysical techniques. A major focus of our research has been on the development of novel membrane mimetics (such as bicelles¹ and nanodiscs) to enable the applications of cutting-edge NMR techniques to study the dynamics structures of membraneassociated peptides and proteins. My lecture will focus on the development of the nanodisc technology for NMR based approaches to study the structural interactions between membrane bound proteins such as cytochromes (~16-kDa b5, ~57-kDa P450, ~80-kDa P450-reductase).²⁻⁴ In addition, strategies to study the structures of membrane stabilized amyloid intermediates will be discussed.⁵⁻⁷ The use of magnetically-aligned nanodiscs to measure residual dipolar couplings (RDCs) and residual quadrupolar couplings (RQCs) will also be presented.

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OXYGEN-17 LABELING AND OXYGEN EXCHANGE KINETICS IN D-FRUCTOSE COMPOUNDS. **Zhonghao Yu** and Gang Wu, Department of Chemistry, Queen's University, Kingston, Ontario, Canada K7L 3N6. (18zy17@queensu.ca)

We report that ¹⁷O NMR can be used to monitor the kinetic oxygen exchange between D-fructose and water. First, $[2-^{17}O]$ -D-fructose was prepared by dissolving D-fructose in $[^{17}O]$ -H₂O and by keeping the solution at 60 °C for several days (the "in-exchange" experiment). The ¹⁷O NMR signals corresponding to different fructose tautomers were observed for the first time. Then "outexchange" experiments were performed where $[2-^{17}O]$ -D-fructose was dissolved in regular H₂O and the water signal increase was monitored over time. The rate of kinetic oxygen exchange was measured as a function of pH between 1 and 11 at 25 °C, which shed light on the oxygen exchange mechanism. In addition, solid-state ¹⁷O NMR results for $[2-^{17}O]$ -D-fructose will be presented.

I130

EXPLORING QUATERNARY CHALCOGENIDE SEMICONDUCTING MATERIALS USING MULTINUCLEAR MAGNETIC RESONANCE SPECTROSCOPY. Vladimir K. Michaelis and Amit Bhattacharya, Department of Chemistry, University of Alberta, Edmonton, Alberta, Canada, T6G 2G2 (vmichael@ualberta.ca)

The decarbonization strategy to shift from legacy fossil fuels to safe and reliable energy alternatives entails the study of highly abundant and inexpensive energy generation and storage materials. One class of semiconducting materials are from a mineral class known as kesterite, Cu₂ZnSnS₄, that are sulfur rich crystalline solids. The challenge in developing these types of materials is to improve upon their stagnant 12% photoconversion efficiency (PCE). The limitation is thought to be attributed to antisite defects (Cu/Zn disorder) due to their similar ionic size and coordination environments which may impact their PCE. This presentation will discuss recent efforts in developing practical approaches to assess Zn/Cu ordering in kesterite and the effect that Cd²⁺- (stannite) and Hg²⁺- (velikite) substitution imparts on their physical properties, local chemical structures and determine the correct crystalline structure ($I\bar{4} vs I\bar{4}2m$). A complementary characterization suite using X-ray diffraction, solid-state nuclear magnetic resonance (NMR) spectroscopy and quantum chemical computations will be presented. A series of multinuclear magnetic resonance spectroscopy measurements will be shown as diagnostic probe nuclei in our efforts to explore the structural evolution directed by M-site substitution and to investigate the antisite Cu/Zn conundrum in a series of high-temperature synthesized solids. We will discuss ^{63/65}Cu MAS NMR results at 21.1 T that show a combination of shielding anisotropy and quadrupolar coupling interactions influenced by their pseudo-tetrahedral environments, while ¹¹⁹Sn NMR reveals distinct second-coordination environments due to Cd/Hg substitution within the M-site. Finally, GIPAW-DFT computations will be discussed to assess potential ionsubstitutions within these quaternary chalcogenide semiconducting materials.

ISOTOPE EFFECTS IN NMR SPECTROSCOPY – A BRIEF OVERVIEW. **Roderick E. Wasylishen**, Department of Chemistry, University of Alberta, Edmonton, AB, T6G 2G2 CANADA. (roderick.wasylishen@ualberta.ca)

The effect of isotopic substitution on the nuclear magnetic shielding and indirect spin-spin coupling constants involving neighbouring nuclei will be reviewed. For example, when a ¹H atom in H₂O is replaced by a deuterium atom, ²H, how does the ¹⁷O nuclear magnetic shielding change as a result of this substitution (i.e., ¹HO²H vs ¹HO¹H)? Also, how is the ¹⁷O, ¹H spin-spin coupling constant changed when one ¹H is replaced by ²H? Several examples of such experiments for samples in the gas or liquid phase will be reviewed and briefly discussed. While the origin of isotope effects is generally well understood, ^{1,2} interpreting such effects in hydrogenbonded systems is much more challenging.³⁻⁶ We will attempt to demonstrate that carrying out NMR measurements on solid samples often has significant advantages over performing similar experiments involving liquid samples. In particular, the examples presented will involve the enol tautomer of 1,3-diketones.

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I132

ADVANCING REPORTING OF SIGNIFICANCE FROM THE ANALYSIS AND COMPARISON OF GLASS EVIDENCE; A GLOBAL COLLABORATION. **José Almirall**, Department of Chemistry and Biochemistry and Center for Advanced Research in Forensic Science (CARFS), Florida International University, Miami, FL 33199, USA. (almirall@fiu.edu)

The results of two interlaboratory exercises involving 20+ operational and research laboratories employing laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) and using a standard test method (ASTM E2927-16e1) for the forensic analysis and comparison of glass evidence are presented. The overall aim of the first exercise was to evaluate the performance of the use of a newly reported calibration standard (CFGS2) designed for microsampling and for the quantitative analysis of simulated glass casework samples. A description of the new glass calibration or non-association (as recommended by the ASTM E2927) and the use of a likelihood ratio (LR) as an objective and quantitative determination of the strength of any association found is also presented. The overall aim of the second exercise was to allow for all participants to gain proficiency in the freely-available Shiny App, to facilitate the calculation of likelihood ratios (LR), compare the likelihood ratios from different scenarios using a wide range of background databases from across the world, and interpret the data to provide recommendations to create a standardized interpretation method for the use of likelihood ratios for glass analysis.

EXPLORING CONFORMATIONAL DYNAMICS OF BIOLOGICAL SUPRAMOLECULAR MACHINES USING METHYL-TROSY NMR SPECTROSCOPY. **Rui Huang**, Department of Chemistry, University of Guelph (rhuang08@uoguelph.ca)

Cellular activities reply on proper functioning of a myriad of large biomolecular complexes. To understand the mechanisms by which these molecular machines work, it is crucial to obtain structural information as well as detailed characterization of their conformational dynamics. Conventional solution NMR is limited to biomolecules with an upper size limit of ~40 kDa; yet, with the development of solution NMR methodology and isotope labeling schemes in the recent decades, we can tackle large biomolecular complexes of up to 1 MDa molecular weight, allowing investigation into challenging biological supramolecular machines. Here we will present examples of using methyl-TROSY NMR to uncover structural and mechanistic details of large biomolecular complexes. We will focus on our previous and on-going work on an AAA+ protein, p97, which is a highly conserved molecular machine involved in a number of cellular functions and has emerged as a promising target for cancer therapy. We have revealed cooperative conformational interconversion of its N-terminal domain and the implication in cofactor binding. We also characterized the complex between p97 and its adaptor protein p47, the complex of which plays an indispensable role in Golgi membrane fusion after mitosis. In addition, we will present our current efforts towards further defining the assembling and subunit exchange process of the p97 complex, as well as probing the mechanisms of two other AAA+ machines, human mitochondrial AAA proteases and bacterial DnaA.

I134

REDUCTION OF MATRIX EFFECTS AND OXIDE INTERFERENCES USING NOVEL LIQUID-LIQUID JUNCTION INTRODUCTION SYSTEM FOR INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY. **Michael Trolio**, Malek Hassan, Richard Oleschuk, Diane Beauchemin. Queen's University Department of Chemistry, 90 Bader Lane, Kingston, ON K7L 3N6, Canada. (<u>17mgat@queensu.ca</u>)

A novel sampling probe composed of a coaxial-tube that functions at a continuous-flow rate was used to inject low volumes of sample $(0.5-2 \ \mu L)$ into inductively coupled plasma mass spectrometry (ICPMS). Simply touching a sample to the dome of liquid at the top of the junction interfaces allows for sample introduction into a flowing solvent stream that transports sample into the plasma - similar to the mechanism of sample transport in flow injection analysis. This self-cleaning sampling system, allows for sampling of microlitres of solids and liquids at atmospheric pressure. Preliminary results suggest a significant reduction of matrix effects and oxide-based interferences. This novel system proved effective in removing matrix effects and interfering species that would typically plague analytical determinations using established sample introduction methods. During method certification using a reference material, our novel method allowed for the correct determination of reference material analytes without the need for an internal standard; unlike when compared to sample introduction using a peristaltic pump.

ENDING BRUTE FORCE PROTEOMICS DATA ACQUISITION USING MACHINE LEARNING. **Mathieu Lavallée-Adam**, Department of Biochemistry, Microbiology, Immunology; Faculty of Medicine; University of Ottawa and Ottawa Institute of Systems Biology. Ottawa, ON, Canada. (mathieu.lavallee@uottawa.ca)

The ability to characterize peptides and proteins using mass spectrometry is linked to data acquisition parameters such as precursor ion selection and collision energy level. Such parameters are often set prior to the mass spectrometry experiment and remain unchanged during the analysis. Application programming interfaces enabling the control of many commercial mass spectrometers allow the modification of data acquisition parameters in real-time during mass spectrometry analysis based on sample characteristics. Machine learning strategies are ideally positioned to support the selection of such parameters to optimize different experimental goals such as maximizing the number of identified and quantified proteins or the fragmentation of precursor ions. To achieve this goal, we present a set of supervised learning algorithms that analyze mass spectrometry data in real-time and use results to further guide data acquisition. We developed a logistic regression classifier that assesses protein identification confidence in real-time and prevents the acquisition of mass spectra from already identified proteins. We also designed an artificial neural network that determines in real-time collision energy levels that will optimize the fragmentation of a given precursor ion. Overall, our methods enhance mass spectrometry proteome characterization capabilities and therefore provide a better understanding of biological processes in analyzed samples.

I136

SEX DETERMINATION OF MUMMIES THROUGH MULTI-ELEMENTAL ANALYSIS OF HEAD HAIR. **Margaret MacConnachie**, Sarah Lu, Yangyang Wang, Diane Beauchemin, Queen's University, Department of Chemistry, 90 Bader Lane, Kingston, ON K7L 3N6, Canada. Jocelyn Williams, Trent University, Department of Anthropology, 1600 West Bank Drive, Peterborough, ON K9L 0G2, Canada. (margaret.macconnachie@queensu.ca)

Sex determination of human remains is of great archaeological significance, as it provides a more complete picture of social and familial structures within ancient societies. Typically performed through examination of bones in the pelvic region, accurate sex determination can be exceedingly challenging in the absence of a sufficiently preserved skeleton. Here, a method for sex determination in living humans, involving measurement of magnesium, strontium, sulfur, and zinc in head hair along with multivariate statistics, was applied for the first time to hair collected from 500-year-old mummies originating from Peru. Using mummy hair as the model enabled accurate sex prediction of the mummies, showing that, despite the age of the hair, the samples still contain the necessary elemental information for sex determination. For accurate sex determination of mummies using hair collected from living humans, magnesium had to be replaced by sodium due to significant differences in dietary habits. With this simple modification, hair from living humans in North America could be used to successfully predict the sex of individuals who lived more than 500 years ago in Peru. This work paves the way for broader use of non-skeletal sex determination methods within the field of archaeology, filling a significant gap.

I137

DETERMINING PHYSICOCHEMICAL PROPERTIES WITH DIFFERENTIAL MOBILITY SPECTROMETRY. W. Scott Hopkins, University of Waterloo, Waterloo, ON, Canada. (scott.hopkins@uwaterloo.ca)

Differential Mobility Spectrometry (DMS) can separate isomeric, conformeric, tautomeric, and even diastereomeric ions based upon differences in their differential mobilities, *i.e.*, the change in ion mobility from low to high electric field conditions. An analyte's differential mobility is determined by how its collision cross section (CCS) changes with the changing electric field and how strongly the analyte interacts with the gaseous collision environment under these dynamic conditions. Consequently, one can modify the collision environment with a low partial pressure of solvent vapor to affect different ion-neutral interactions, thereby altering differential mobility and, in many cases, improving analytical separation. We have demonstated that degree to which differential mobility changes upon adding a solvent modifier correlates with the strength of the ion-solvent interactions. However, different classes of molecules exhibit slightly different trends owing to variations in size, shape, and interaction potentials. Using machine learning (ML), we can treat a wide variety of analytes simultaneously and create predictive models for molecular properties including CCS, solubility (Log S), distribution co-efficient (Log P), pK_a, and passive cell permeability. In this presentation, I will discuss our most recent results and provide a perspective for future research directions.

I138

ICP-MS FOR SINGLE CELLS ANALYSIS. **Bin Hu**. Department of Chemistry, Wuhan University, Wuhan, 430072 (<u>binhu@whu.edu.cn</u>)

ICP-MS is a sensitive detection technique for monitoring of metal/metalloids in single cell. However, there are still some problems such as multiple cellular events for one signal spike in single cell analysis by pneumatic nebulization-ICP-MS. Microfluidic chip is suitable for cell manipulation, either encapsulation of single cell in droplets or ordering cells into a single stream, which facilitates subsequent single cells detection.

In this presentation, the potential of microfluidic chips combined with ICP-MS for single cell analysis will be illustrated with some examples [1-5].

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I139

TRANSIENT INCOMPLETE SEPARATION OF SPECIES WITH CLOSE DIFFUSIVITY TO STUDY STABILITY OF AFFINITY COMPLEXES. Tong Ye Wang,^a Jean-Luc Rukundo,^a An T.H. Le,^a Nikita A. Ivanov,^a C. Yves Le Blanc,^b Boris I. Gorin,^c and **Sergey N. Krylov**^a ^aDepartment of Chemistry and Centre for Research on Biomolecular Interactions, York University, ^bSCIEX, ^cEurofins CDMO Alphora. (skrylov@yorku.ca)

Large molecules can be generically separated from small ones, though partially and temporarily, in a pressure-driven flow inside a capillary. This transient incomplete separation has been only applied to species with diffusion coefficients different by at least an order of magnitude [1–5]. Here, we demonstrate, for the first time, analytical utility of transient incomplete separation for species with close diffusion coefficients. First, we prove in silico that even a small difference in diffusivity can lead to detectable transient incomplete separation of species. Second, we use computer simulation to prove that such separation can be used for reliable determination of equilibrium dissociation constant (K_d) of complexes composed of similar-size molecules. Finally, we demonstrate experimentally the use of this separation for accurate determination of K_d value for a protein-aptamer complex. We conclude that "accurate constant via transient incomplete separation" (ACTIS) can serve as a reference method for affinity characterization of protein-aptamer binding in solution.

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I140

ANALYTICAL CHEMISTS SAVING UNIQUE 19TH CENTURY GLASS ARTIFACTS. Ela Bakowska, Corning RDC, SP-FR-06, Corning NY, 14831, USA, (<u>bakowskae@corning.com</u>)

Leopold and Rudolph Blaschka (father and son team) are mostly known for 3000+ collection of botanical specimens' glass models they made exclusively for the Harvard Botanical Museum. Prior to botanical glass model, from 1863 to 1890, they were making glass models of marine invertebrates in their studio in Dresden, Germany. Multiple museums and universities acquired those models for educational purposes. Even today researchers at Cornell continue to study them. The Blaschka marine models were made to scale and sometimes close to life size, resulting in dimensions from 1 to 27 cm. Nearly all glass used in their creation was colorless. A collection of damaged marine invertebrates' models was found University of Wisconsin. Many of the models were in a terrible state of preservation, but some were intact enough to put on display. Corning RDC was approached to help curate the damaged models. Once determined (using ICP-OES, FAE and ICP-MS) the original glass' compositions, then the historically accurate replica glasses can be re-created and used to repair the damaged models. We determined and evaluated 65 elements in 14 glasses. Nine models were from Cornell University / Corning Museum of Glass (CMoG) collection and five glasses from damaged UW/Madison collection.

RAPID AND AUTOMATED ELEMENTAL SPECIATION FOR CLINICAL, ENVIRONMENTAL, AND FOOD APPLICATIONS. **C Derrick Quarles Jr.**¹, Patrick Sullivan¹, Nick Bohlim¹, Marcel Macke², Andrew Toms¹, Uwe Karst². ¹Elemental Scientific, Inc., Omaha, NE, USA; ²University of Munster, Munster, Germany. (<u>Derrick.quarles@icpms.com</u>)

Understanding the specific chemical species found in different samples is important to toxicological, clinical, environmental, food, pharmaceutical, and geochemistry. Arsenic is found in many forms in the natural environment, some of which are significantly more toxic than others. Therefore, fast and reliable analytical methods are required to determine which arsenic species are present. We present two methods for determining arsenic species in urine by LC-ICP-MS. The first method is a high-throughput method for separating 6 arsenic species (AsB, DMA, MMA, AsC, As(III), and As(V)) in under 2 minutes. The second method provides a separation of 7 arsenic species (AsB, TMAO, DMA, MMA, AsC, As(III), and As(V)) in under 5 minutes. The validation of both methods was carried out by analyzing proficiency testing (PT) samples from the Centre de Toxicologie du Québec (CTQ) and New York Department of Health (NYDOH). Recovery of the two methods falls between 94 and 107% and the limits of detection in a urine matrix ranged from 2.8-6.0 ng L⁻¹ As and 4.1-9.1 ng L⁻¹ As for the one- and two-column methods, respectively. In addition, the determination of Gd based contrast agents has been automated for environmental samples collected from surface waters. This automated method shows excellent correlation between the total inorganic Gd and the Gd based contrast agents determined by LC-ICP-MS.

I142

MICROWAVE DIGESTION AND ANALYSIS OF 10 DIFFERENT FOOD SAMPLES PREPARED IN A SINGLE BATCH. **Bob Lockerman**, CEM Corporation, USA. (Bob.Lockerman@cem.com)

The US Food and Drug Administration (FDA) regulates food testing for the US and many other countries. The FDA publishes Elemental Analysis Methods (EAM) which provides procedures governing food testing. In August 2020 EAM 4.4 and 4.7 prescribed the conditions for the digestion and analysis by ICP-OES and ICP-MS. Microwave digestion conditions are prescribed as a part of the EAM. Microwave digestion of food materials have been done for decades. In the past foods had to digested by food types. This meant they had to be separated into categories for foods high in carbohydrates, proteins and fats. Samples then had to be digested in separate batches which is inefficient especially for contract laboratories who need to provide a low cost per test to provide competitive pricing. Advances in temperature control and vessels have now made it possible to prepare these samples all together in a single batch. It allows for up to twenty four samples to be prepared and ready for dilution and analysis in less than 45 minutes. Subsequent sample racks can be prepared so that once the first batch is processed a second can be placed in the microwave system providing a highly efficient means to process food samples for metals analysis. In this presentation we will use a MARS 6 digestion system and a simple to use 3 part MARSXpress Plus vessel to prepare 10 different types of foods following EAM 4.4 and 4.7 methodology. We will show analytical results to confirm that microwave digestion of mixed food types in a single run is accurate as well as efficient

I143 P

COMPARISON OF LOW VOLUME INJECTION TO MORE CONVENTIONAL SAMPLE INTRODUCTION IN INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY FOR ITS EFFECT ON OXIDE FORMATION AND MATRIX EFFECTS. **Graham Shearing**, Michael G.A. Trolio, and Diane Beauchemin. Queen's University, Department of Chemistry, 90 Bader Lane, Kingston, ON K7L 3N6, Canada. (diane.beauchemin@queensu.ca)

Oxide interferences and matrix effects are known to compromise the accuracy of inductively coupled plasma mass spectrometry (ICPMS) measurements. A method to consistently reduce the impact of these interferences on empirical results would be a valuable tool for both research and industry. A new sample introduction method was explored through parallel experiments targeted at examining oxide formation and matrix effects. Samples were introduced using a monosegmented (MS) flow injection analysis (FIA) technique with 1 microlitre of sample. This technique was compared to both peristaltic pump and 50 microlitre FIA sampling methods. Oxide formation was examined through measuring CeO+ /Ce ratios while injecting a solution of 10 ppm Ce. A positive correlation between volume and interference was observed and the 1 µL MS-FIA offered up to a 68.8% reduction in CeO+ /Ce. Matrix effects were examined across 12 elemental solutions of 10 ppm, with and without 400 ppm of Na. The average changes in signal with the addition of Na for peristaltic, 50 µL FIA, and 1 µL MS-FIA were -4.7%, -1.11%, and 0.18% respectively. Finally, CRM analysis was performed using calibrations without internal standard, for all three techniques. A negative correlation between sample volume and accuracy was observed and the 1 µL MS-FIA technique was the only one to yield concentrations within the range of inclusion for all successfully observed elements. The results indicate that both oxide interferences and matrix effects can be reduced using low volume MS-FIA and these reductions translate into overall increases in measurement accuracy.

I144 P

SINGLE SPOT ANALYSIS OF PARAFFIN-EMBEDDED TISSUE SECTIONS USING LIQUID MICROJUNCTION SAMPLING PROBE (LMJ-SSP) MASS SPECTROMETRY (MS). **Haidy Metwally**, Malek Hassan, Jessie Deng, and Richard Oleschuk, Department of Chemistry, Queen's University, Kingston, ON, K7L 2S8. (hm87@queensu.ca)

Freezing fresh tissues is the optimum method for conserving nucleic acids and proteins. However, fresh frozen (FF) tissues require strict storage conditions such as -80 temperature for up to one year. In addition, FF tissues suffer from the formation of ice crystals, and they became fragile with time. Formalin fixation and paraffin embedding (FFPE) of tissues improve their processing and extend their shelf life. FFPE tissues are an important source of archived biological data. FFPE tissues cannot be used directly with mass spectrometry imaging as paraffin needs to be removed. The protocol used to remove the paraffin from the tissues usually starts with exposing the FFPE tissue to xylene, followed by washing with serial dilutions of ethanol. This study aims to decrease the amount of time spent on tissue cleaning and processing as well as preserve the tissue for further storage. Using the LMJ-SSP mass spectrometry; we were able to dewax certain spots on the FFPE chicken liver and detect some bile acid signals.
ATOMICALLY PRECISE METAL CLUSTERS: SYNTHESIS, UNIQUE PHOTOPHYSICAL PROPERTIES, AND THEIR ADVANTAGES IN BIOMEDICAL APPLICATIONS. **Goonay Yousefalizadeh**¹, Parimah Aminfar¹, Elham Zeinizade^{2,3}, Juan Chen³, Kevin Stamplecoskie¹, ¹Queen's University, Department of Chemistry, 90 Bader Lane, Kingston, ON K7L 3N6, Canada; ²Department of Medical Biophysics, University of Toronto, Toronto, ON M5G 1L7, Canada; ³Princess Margaret Cancer Centre, University Health Network, Toronto, ON M5G 1L7, Canada. (yousefalizadeh@queensu.ca)

Atomically precise metal clusters, like the extensively studied [Au25SR18], are a class of extremely small (< 2 nm) metal nanomaterials. Clusters have exact numbers of metal atoms and organic ligands and structures that have been completely determined by mass spectrometry and X-ray crystallography. Due to the extremely small size of these clusters, they exhibit molecule-like electronic and optical properties that are highly tunable. The unique photophysical properties of clusters make them great candidates for multiphoton biomedical imaging and photocatalysis. In this talk I will discuss our approach to isolating pure clusters. I will discuss the ultrafast nump/probe spectroscopy studies that provide deep insight into the excited state behaviour of

pump/probe spectroscopy studies that provide deep insight into the excited state behaviour of clusters, which plays an important part in dictating the photocatalytic activity. Also presented will be results on two photon absorbance (2PA) cross sections of gold and silver clusters which is used to evaluate the structure-dependent non-linear optical properties of a series of thiol stabilized gold and silver cluster. Lastly, some surprising results will be presented related to the potential of these materials as new generation of photonic materials in cancer therapy applications.

I146

SYNCHROTRON X-RAY SPECTROSCOPY OF SOME METAL NANOCLUSTERS. Peng Zhang. Department of Chemistry, Dalhousie University, Halifax, NS B3H 4R2, Canada. (Peng.Zhang@Dal.ca)

Synchrotron X-ray spectroscopy techniques are powerful tools for the atomic scale analysis of structure and properties of materials. In this talk, the application of these X-ray techniques in the study of some noble metal nanoclusters will be presented. It will be demonstrated that the X-ray spectroscopy methods can sensitively probe the structure and physicochemical properties of the metal nanostructures at the atomic scale, and thus help more thoroughly understand their structure-property relationships. The physical insights into these nanostructures provided by the synchrotron techniques should also be useful in guiding their catalytic and biomedical applications.

I147 P

NANO-GOLD OF VARIED SIZE AND COMPOSITION: A THEORETICAL X-RAY SPECTROSCOPY ANALYSIS. Lydia Zhang^{1,2}, Peng Zhang¹, Michael Ha². ¹Department of Chemistry, Dalhousie University, Halifax, NS, B3H4R2, Canada; ²Department of Radiation Oncology, Dalhousie University, Halifax, NS, B3H1V7, Canada. (Peng.Zhang@Dal.ca)

Gold-based nanomaterials are promising materials for various biomedical applications. Understanding the structure-property relationship of nano-gold at atomic scale plays an important role toward developing high-performance functional materials towards these applications. In this poster presentation, a series of magic number gold nanoclusters with varied size and alloying composition is studied by theoretical X-ray absorption spectroscopy. It is found that gold atoms at specific atomic sites such as vertex, edge and terrace surface site show significantly different bonding properties. When the size of the nano-gold is varied, the average electronic and bonding properties are found to be highly dependent on the cluster size and coordination environment. Finally, silver-gold alloy nanoclusters with well-defined core-shell structure are investigated and bonding properties of the silver-gold samples are compared with their gold counterparts to demonstrate the alloy effect on the structure and properties of nano-gold. These findings will be useful to better understand the structure-property relationship of nano-gold from the perspective of atomic site, cluster size and alloy bonding.

I148

STRATEGIES AND APPROACHES FOR PARTICULATE MATTER (PM) ANALYSIS IN AIR. Chady Stephan, **Ruth Merrifield**, Aaron Hineman. PerkinElmer Inc., #6, 501 Rowntree Dairy Rd, Woodbridge, ON, Canada. (Chady.Stephan@PERKINELMER.com)

Atmospheric aerosols, or particulate matter (PM) are one of the main sources of climate change. However, the mixing state of aerosols and the influence of particle composition on particle size isn't well known. These information's are lost when PM are analyzed for metal content after filter collection, thus the need for direct analysis.

In this talk, we will review a method that has been developed to directly analyze these aerosols on a single particle basis using a gas-exchange device (GED) coupled with quadrupole inductively coupled plasma-mass spectrometry (ICP-MS). We will discuss strategies and approaches to screening for PM in air using GED-ICP-MS. We will also discuss the ability of screening for multielement on a particle basis using a quadrupole based ICP-MS.

ILLUMINATING THE EDGE OF DISCOVERY, NEOMA MULTICOLLECTOR ICP-MS. **P. Stow**¹, C. Bouman², G. Craig², J. Roberts², N. S. Lloyd², M. Pfeifer², H. Wehrs², D. Tollstrup², J. Schwieters. ¹ Isomass Scientific Inc., Calgary, Alberta; ² Thermo Fisher Scientific, Hanna-Kunath Str. 11, 28199 Bremen, Germany. (pstow@isomass.com)

Multicollector inductively coupled mass spectrometry (MC-ICP-MS) has become the workhorse of isotope ratio mass spectrometry since its introduction 25 years ago. The full application of MC-ICP-MS has been limited however by some problematic, mainly isobaric, interferences. Collision/reaction cell (CRC) technology can be advantageous in MC-ICP-MS as it enables reactions of specific elements with the reacting gas, thereby creating a mass difference between the element of interest and the isobaric interference. In single collector ICP-MS, collision/reaction cell technology has been combined with a mass filter in front of the cell. The pre-cell mass filter eliminates the issue of secondary reactions in the collision/reaction cell. This MS/MS technology has been successfully applied for a variety of exciting geoscience applications [1]. Many of these ICP-MS/MS applications would benefit from the excellent isotope ratio precision available from MC-ICP-MS. Additionally, adding MS/MS capability to a MC-ICP-MS would significantly enhance its use and open up various new applications that cannot be explored with classical MC-ICP-MS technology. Here we report on the new MS/MS capability of the Neoma MC-ICP-MS and its benefit for a variety of new applications within geosciences. The Neoma MC-ICP-MS can be upgraded to full MS/MS, adding a novel pre-cell mass filter and hexapole collision/reaction cell [2]. The resulting MC-ICP-MS/MS requires no compromises in either sensitivity, accuracy or precision. With the ability to select a discrete range of masses to enter the collision/reaction cell [2]. SF₆ and NH₃ can be used alongside H₂ and He.

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[2] Craig, G.; Wehrs, H.; Bevan, D. G.; Pfeifer, M.; Lewis, J.; Coath, C. D.; Elliott, T.; Huang, C.; Lloyd, N. S.; Schwieters, J. B. Anal. Chem. 2021, 93, 10519.

I150

ANALYTICAL CONSIDERATIONS FOR THE USE OF SP ICP-MS WITH NATURAL SAMPLES. Ibrahim Jreije, Agil Azimzada, Madjid Hadioui, **Kevin J. Wilkinson**. Department of Chemistry University of Montreal, CP 6128, succ. Centre-ville, Montréal, QC, H3C 3J7, Canada. (kj.wilkinson@umontreal.ca)

With the significant increase in the production and use of nanoparticles (NP), concern is increasing over their release into their environment. Single particle inductively coupled plasma mass spectrometry (SP-ICP-MS) is emerging as one of the best techniques for detecting the very small NP at very low concentrations in natural waters. However, there is no unified protocol for the preparation of natural water samples for SP-ICP-MS analysis. In order to minimize nebulizer blockage, filtration is often used with the expectation that 0.45 μ m membranes will not remove significant quantities of 1-100 nm NP. Nonetheless, there are limited data on its effect on the concentrations or size distributions of the NP. To that end, we examined the interactions between six different membrane filters and silver (Ag) and cerium oxide (CeO₂) NP in aqueous samples. For Ag NP, the highest recoveries were observed for polypropylene membranes, where 55% of the pre-filtration NP were found in rainwater and 75% were found in river waters. For CeO₂ NP, recoveries could be increased to over 80% by pre-conditioning the filtration membranes with a multi-element solution. Similar recoveries were obtained when samples were centrifuged at low centrifugal forces ($\leq 1000xg$).

CUSTOMIZABLE SILVER NANOPARTICLE SERS FILMS: OPTIMIZATION AND PESTICIDE SENSING. **Graham Beaton**¹, Yazan Bdour², Carlos Escobedo², Kevin Stamplecoskie¹. ¹Department of Chemistry, Queen's University; ²Department of Chemical Engineering, Queen's University, Kingston, ON K7L 3N6, Canada. (16gb17@queensu.ca)

Silver nanoparticles have been widely researched and celebrated in their ability to perform surface enhanced Raman spectroscopy for chemical sensing. Despite many advantages to silver over other plasmonic metals, the reactivity and unwanted chemistry common for silver makes substrate fabrication challenging, leaving the true potential of silver underutilized for many applications. silver nanoparticles supported on alumina, showcase robust stability while remaining labile to photochemical shape control methods. These easily reproduced films can be tailored across the visible spectrum depending on user needs and show long term performance as SERS sensors. Optimization parameters are explored, and these films are showcased as promising chemical sensors for the pesticide thiram.

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INNOVATIVE AND MODERN SOLUTIONS FOR SAMPLE PREPARATION PRIOR TO ICP ANALYSIS. Adam Abdelrehim, Anton Paar Canada, 4920 Place Olivia, Montreal, QC H4R 2Z8. (adam.abdelrehim@anton-paar.com)

Superior trace elemental analysis begins with outstanding sample preparation. With over 40 years of experience, Anton Paar strives to make microwave digestion easier and more convenient than ever before – thanks to sophisticated instrument and vessel concepts, state-of-the-art sensor technology, and the highest safety standards. Anton Paar offers the broadest range of instruments for all kinds of applications: microwave digestion systems Multiwave 7000 and Multiwave GO Plus, the microwave reaction platform Multiwave 5000 and conventionally heated Multicube 48. Choosing the right system for your sample has never been easier. Each microwave system can be configured to prepare samples of varying complexities. Tool-free vessel designs streamline the process even further. In this presentation, several applications with results will be presented including digestion of cannabis materials, battery raw materials, and environmental samples.

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TWO PHOTON ABSORBANCE AND ACTION SPECTRUM OF SILVER CLUSTERS FOR PHOTODYNAMIC THERAPY. **Emily Steele**, Rachel Odell, James Fraser, Kevin Stamplecoskie, Queen's University, Kingston, ON K7L 3N6, Canada. (16eas1@queensu.ca)

Photodynamic therapy is a leading clinical method for cancer treatment with the advantage of being minimally invasive. The majority of the therapeutic agents in this treatment use visible light irradiation to trigger cell death, however, visible light has very low penetration in human tissue, thereby limiting the types of cancers that photodynamic therapy can treat. This presents a unique opportunity: since the photosensitisers clinically available today are all organic dye molecules, they require excitation wavelengths in the visible range, which has relatively low transmission into human tissue (max 1 cm), especially when compared to infrared light. Using a material like metal nanoclusters would allow for absorption of deeper penetrating light, expanding the scope of cancers that can be treated with photodynamic therapy by studying the excited state dynamics and two-photon absorbance.

I154

APPLICATION OF HIGH-RESOLUTION ANALYTICAL TECHNIQUES FOR DETECTION OF PFAS IN COMMERCIAL PRODUCTS. **Taylor Vereecken**, David Patch, Natalia O'Connor, Iris Koch, Kela Weber. Environmental Sciences Group, Department of Chemistry and Chemical Engineering, Royal Military College of Canada, Kingston, ON, Canada K7K 7B4. (tvereecken5@gmail.com)

Per- and polyfluoroalkyl substances (PFAS) are commonly used in many industrial and commercial products to take advantage of their unique physicochemical properties. However, these properties also make PFAS recalcitrant, and have resulted in widespread contamination of the environment and human population. Humans are exposed to PFAS mainly through ingestion, but recent research has focused on the risk of PFAS in cosmetic products. Through the use of cutting-edge analytical techniques, including high resolution mass spectrometry (HR-MS), total oxidizable precursor assay (TOPA), and a total reducible organic fluorine assay (TROFA), the total organofluorine content present in cosmetic makeups is determined. A wide assortment of PFAS were identified in cosmetic products tested, suggesting dermal sorption of PFAS may present a wider risk to people than previously understood. This investigation provides transparency of the toxicity of daily products and the need for safer products.

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N-HETEROCYCLIC CARBENES AS LIGANDS FOR METAL SURFACES AND NANOCLUSTERS **Cathleen Crudden**^{1,2}. ¹Department of Chemistry, Queen's University, Kingston, Ontario, K7L 3N6 Canada; ²Institute of Transformative Bio-Molecules, Nagoya University, Nagoya, Japan. (cruddenc@chem.queensu.ca)

The use of N-heterocyclic carbenes to modify homogeneous metal catalysts is widespread since the high metal–NHC bond strength renders high oxidative and chemical stability to the resulting metal complexes. The use of NHCs to modify metal surfaces has received considerably less attention. We will describe the modification of planar metallic surfaces with NHCs. The nature of the surface overlayer is strongly dependent on the nature of the NHC. Similarly, NHCs are useful new ligands for the stabilization of metal clusters, with the structure of the cluster being strongly influenced by the nature of the NHC. The physical and photophysical properties of these clusters will be addressed.



I156

DEMOCRATIZING NMR SPECTROSCOPY: INTRODUCTION TO NANALYSIS BENCHTOP NMR AND APPLICATIONS. **Matthew T. Zamora**, Nanalysis, Canada. (matt.zamora@nanalysis.com)

Traditional Nuclear Magnetic Resonance (NMR) instrumentation, although an extremely powerful characterization tool in research, has been largely excluded from a variety of applications – e.g., academic teaching, industrial QA/QC, process analytical technology (PAT). The realities of the super conducting NMR footprint and the required routine maintenance, paired with their expensive capital and operating expenditures have left this non-destructive, inherently quantitative technique limited. The recent developments of a new class of instrumentation, the compact, portable, benchtop NMR spectrometer, offers an interesting alternative to democratize NMR. Herein we will introduce the benchtop NMR product line, discuss some application development, and highlight use cases as we look to expand the use of NMR Spectroscopy outside of traditional structure elucidation type applications.

COMPREHENSIVE TARGETED AND NON-TARGETED LIPIDOMICS ANALYSES IN FAILING AND NON-FAILING HEART. Ganesh V. Halade¹, Anela Dorbane², Kevin A. Ingle¹, Vasundhara Kain¹, Jean-Marie Schmitter² and **Boutayna Rhourri-Frih²**. ¹Division of Cardiovascular Disease, Department of Medicine, The University of Alabama at Birmingham, 703 19th Street South, MC7755, Birmingham, AL, 35294, USA; ²Chimie et Biologie des Membranes et Nanoobjets, University of Bordeaux, CNRS UMR 5248, 146, rue Léo Saignat, 33076, Bordeaux, France.

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THERMO SCIENTIFIC VANQUISH SYSTEMS- SEE PRODUCTIVITY IN A NEW LIGHT. Vikas Chander. Thermo Fisher Scientific.

Everyday labs are challenged with increased workload and less resources. Thermo scientific unique solutions solves this challenge by some of the unique workflows which yields more productivity, increased utilization of benchspace, instruments and in a under friendly manner.