



# ***EnviroAnalysis* 2015**

**The Environmental Solutions Conference**

**July 12-15, 2015  
The Banff Centre  
Banff, Alberta**

## **Program**

# Exhibitors and Sponsors

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**Agilent Technologies**



# Welcome to Delegates

Dear Colleagues:

Welcome to **EnviroAnalysis 2015**, the Tenth Environmental Solutions Conference. We know that competition for your conference funds is intense, so we have worked hard to ensure you receive good value for your money. The important features of this year's conference are:

- Focussed meeting: Delegates to **EnviroAnalysis 2015** have access to recent results from many areas of the analytical / environmental community.
- Expanded scope of meeting: for the first time, EnviroAnalysis is open to papers in *all areas* involved with describing and improvement of the natural environment.
- Top international plenary and keynote speakers: organizers of **EnviroAnalysis 2015** have increased the number of international speakers in a wide variety of environmental analysis disciplines.
- Laboratory Exhibit: Exhibitors of interest to the environmental analytical community will be on hand for two days to discuss the latest advances in supplies and equipment.

This conference has always been organized in response to the needs and wishes of the delegates. Thus we are interested in hearing from you about how to make the meeting better the next time. Please send us your suggestions for **EnviroAnalysis 2017**, to be held in Toronto, Ontario, Canada (new date Spring 2017): [ray.clement@enviroanalysis.ca](mailto:ray.clement@enviroanalysis.ca).



Teresa Switzer, Ontario Ministry of Environment and Climate Change  
**EnviroAnalysis 2015** chair



Ray Clement, President, EnviroAnalysis  
Conference Series Organizer



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# General Conference Information

## Dress Code

Dress code for the conference will be "Mountain Casual".

## Breakfasts and Lunches

Breakfasts are provided for all registered delegates Monday, Tuesday and Wednesday in the Vistas Dining Room from 7:00 – 8:30 AM. Lunches Monday and Tuesday are in the Vistas dining room 11:45 AM to 1:20 PM. Delegates access the Vistas Dining Room with their hotel keycard.

## Karasek Award

Wellington Laboratories has generously donated the funds for student poster awards. One student will earn recognition based on his/her scientific contribution and poster presentation. The award will be given out at the conference banquet.

## The Founders' Award

Established in 2002 by the founders of *EnviroAnalysis*, this award recognizes the significant contributions of industry and government organizations for the support of environmental education, communication, research and technology transfer activities. This year, Alberta Innovates – Energy and Environment Solutions and Alberta Innovates – Technology Futures will be recognized.

## Banquet

The conference banquet will take place Tuesday, July 14 from 6:00 – 10:00 PM. Full conference registered delegates and those who paid the "accompanying person" fee are all invited at no additional cost. Others must purchase banquet tickets Monday morning if they have not done so beforehand.

Monday Morning, July 13  
Opening Plenary Session  
Kinnear Centre (KC) 201

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8:30	Welcome to Delegates	Jim Olver Director of Customer Services The Banff Centre
8:40	Opening Remarks	Teresa Switzer <i>EnviroAnalysis</i> 2015 Chair Dr. Ray Clement <i>EnviroAnalysis</i> Series Organizer
9:00	Anatomy of an Oil Spill and its Subsequent Chemical Evolution by Ultrahigh-Resolution Fourier Transform Ion Cyclotron Resonance Mass Spectrometry (Abstract 13)	Dr. Alan Marshall Florida State University
9:45	Uncertainty, Bias, and Error in Environmental Measurement and Risk Determination (Abstract 55)	Dr. Murray McBride Cornell University
10:30	Refreshment Break <i>KC 201/Foyer</i>	
11:00	The Founders Award Alberta Innovates: Technology Futures Alberta Innovates: Energy and Environment Solutions	Dr. Ray Clement
11:05	Environmental Human Health: Exposome and Epigenome	Dr. H.A. (Skip) Kingston Duquesne University
11:50	Lunch <i>Vistas Dining Room</i>	

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## Monday Afternoon, July 13

### Persistent Organic Pollutants (POPs) Analysis KC 201

Session Chair: Ray Clement

### Industrial Applications KC 205

Session Chair: Vasili Furdui

<p><b>Keynote:</b> Donald G. Patterson, Jr., EnviroSolutions Consulting, Inc. <i>Persistent Organic Pollutants--The External Versus Internal Human Dose Measurement</i></p>	1:20	<p><b>Keynote:</b> Graeme Spiers, G. Koptsik, S. Koptsik, P. Beckett and V. Udachin <i>The Environmental Legacy of Metal Smelters on Northern Landscapes</i></p>
<p>Dave Hope [11] <i>Inexpensive Rapid Method for Pops Analysis of Food Using ASE and Tandem Acid Silica/Carbon Columns</i></p>	2:00	<p>William Shotyk [54] <i>Peat cores from ombrotrophic (rain-fed) bogs in northern Alberta reveal decreasing atmospheric contamination by Pb and other heavy metals</i></p>
<p>Cassandra Rauert, Lutz Ahrens, Mahiba Shoeib, Tom Harner [35] <i>The occurrence of HBCD and its diastereomers in Toronto urban and global air</i></p>	2:20	<p>Joachim Weiss [41] <i>Reagent-Free Ion Chromatography (RFIC) and its Application in Environmental Ion Analysis</i></p>
<p><b>2:40 Exhibitor Viewing and Refreshments KC 203</b></p>		
<p>Haiyan Helena Wang, Graham Knox, Julius Pretorius [28] <i>Determination of Volatile Organic Compounds in Ambient Air by Gas Chromatography Mass Spectrometry</i></p>	3:40	<p>Chad W. Cuss, William Shotyk, Ehsanul Hoque, Beatriz Bicalho [45] <i>Characterizing the size and optical properties of Athabasca R. dissolved organic matter: towards bitumen-based optical tracers and measurement of metal partitioning</i></p>
<p>Alexander Semyonov, Dwain Cardona, Amit Gujar [38] <i>Analysis of Pesticides and Dioxins in Food on Modern Triple Quad GC-MS/MS</i></p>	4:00	<p>Rob Taylor, Teresa Switzer, Darko Ilic, Shulan Liu [44] <i>The Application of ICPMS in Nuclear Industry - Determination of Pu Isotopes in Urine Sample</i></p>
<p>Dave Hope [12] <i>Evaluation of TSQ8000 (GC-MS/MS) for PCDD/F Analysis</i></p>	4:20	<p>Vinson Leung, Michael Ebitson, Jim Fenster, Zoe Grosser, Toni Hofhine, Jean-Luc Brousseau [49] <i>Maintaining the Power Grid - a Simplified and Efficient Approach to Routine Monitoring of Transformers Using Automated Solid Phase Extraction Furan Testing in Transformer Oil</i></p>
<p>Paul Fayad, Audrey Roy-Lachapelle, Sung Vo Duy, Michele Prevost, Sebastien Sauve [14] <i>On-line solid-phase extraction coupled to liquid chromatography tandem mass spectrometry for the analysis of cyanotoxins in algal blooms</i></p>	4:40	
<p><b>5:00 – 6:30 Exhibitor Wine and Cheese Reception Sponsored by Thermo Fisher Scientific KC 203</b></p>		

## Tuesday Morning, July 14

### Environmental Impacts of Oil Sands Development *KC 201*

Session Chair: Jon Fennell

### Spectroscopy

*KC 205*

Sponsored by PerkinElmer

<p><b>Keynote:</b> <u>Kevin Percy</u>, Wood Buffalo Environment Association title tba</p>	8:40	<p><u>Pamela Wee</u> [1] <i>The Direct Analysis of Multiple Elements in 25% Brines using an Agilent 7900 ICP-MS with an Ultra High Matrix Interface</i></p>
	9:00	<p><u>Xinbang Feng</u>, Lisa Shi, Julius Pretorius [3] <i>Determination of Total Mercury in Fish Muscle Samples by Inductively Coupled Plasma Mass Spectrometry</i></p>
<p><u>Alberto dos Santos Pereira</u>, James Srorey, [29] Graham Knox, Julius Pretorius <i>Quantification of Naphthenic Acids in Water Samples: Challenges for complex matrix?</i></p>	9:20	<p><u>Aaron Hineman</u>, Chady Stephan [19] <i>Single Particle ICP-MS (SP-ICP-MS) for the Detection of Metal-Based Nanoparticles in Environmental Matrices</i></p>
<p><u>Yi Yi</u>, Jean Birks, Sunny Cho, John J. Gibson [51] <i>Characterizing organic compositions in snow and surface water in the Athabasca oil sands region</i></p>	9:40	<p><u>Pamela Wee</u> [2] <i>Implications of Performance Metrics on the Quality of ICP-MS Analysis</i></p>
<p><u>Jonathan Martin</u>, Chenxing (Angela) Sun [24] <i>Differentiating Natural and Anthropogenic Sources of Bitumen-Derived Organic Compounds to Water Resources Surrounding the Canadian Oil Sands Industry</i></p>	10:00	<p><u>Lisa Shi</u>, Xinbang Feng, Julius Pretorius [4] <i>Microwave Digestion of Biological Tissue Standard Reference Materials for Multi-elemental Determination by ICP-MS</i></p>
<p><b>10:20 Refreshments</b> <i>KC 203</i></p>		
<p><b>11:00 - Plenary</b> <i>KC 201</i> <u>Jonathan Martin</u> <b>Investigating Known Unknowns in the Supercomplex World of Oil Sands Process Water</b> (Abstract 56)</p>		
<p><b>11:45 Lunch</b> Sponsored by PerkinElmer <i>Vistas Dining Room</i></p>		

Tuesday Afternoon, July 14

**Environmental Impacts of Oil  
Sands Development  
KC 201**

Session Chair: Jon Fennell

**Mass Spectrometry:  
Techniques & Applications  
KC 205**

Session Chair: Susan Richardson

<u>Keynote: Tom Harner</u> [34] <i>Polycyclic Aromatic Compounds (PACs) in Air across the Athabasca Oil sands region: Assessing Ecosystem Exposure</i>	1:20	<u>Keynote: Michael Ketterer</u> [42] <i>A retrospective look at Nevada Test Site fallout: studies with plutonium isotopes</i>
<u>Narumol Jariyasopit, Sabina Halappanavar, Tom Harner</u> [30] <i>Toxicity Mapping using Passive Air Sampling for Polycyclic Aromatic Compounds</i>	2:00	<u>Donald G. Patterson, Jr., EnviroSolutions Consulting, Inc.</u> <i>Mass Spectrometry and Biological and Environmental Forensics: Parts Per Quintillion--A New Frontier for Toxicologists and Risk assessors</i>
<u>Jason Ahad, Josué Jautzy, Anna Smirnoff, Charles Gobeil, Martine Savard</u> [6] <i>An isotopic approach to source apportionment of PAHs in the Athabasca oil sands region</i>	2:20	<u>Marcus Kim, Dayue Shang, Maxine Haberl, Jeffrey Yan</u> [22] <i>Rapid And Sensitive Method For The Determination Of Polycyclic Aromatic Hydrocarbons In Soils Using Pseudo Multiple Reaction Monitoring Gas Chromatography/ Tandem Mass Spectrometry</i>
<b>2:40 Poster and Exhibitor Viewing and Refreshments KC 203</b>		
<u>Gillian Mullan-Boudreau, William Shotyk</u> [39] <i>Moss and peat as monitors of past, present, and future rates of atmospheric dust deposition</i>	3:40	<u>Susan Richardson, Cristina Postigo, Christina Joseph, Friedrich Wendel, Christian Luetke-Eversloh, Thomas Ternes, Edward Machek, Stephen Duirk, Elizabeth Wagner, Michael Plewa</u> [9] <i>Tandem Mass Spectrometry and High Resolution Mass Spectrometry Uncovers Chlorination Reaction Pathway for X-Ray Contrast Media in Drinking Water Treatment</i>
<u>Hui Peng, Hattan Harbi, Jianxian Sun, Steve Wiseman, Paul Jones, John P. Giesy</u> [17] <i>A non-targeted chemical analysis strategy for identification of dissolved organic chemicals that cause acute toxicity of oil sands process-affected water</i>	4:00	
<u>Beatriz Bicalho, Iain Grant-Weaver, Gillian Mullan-Boudreau, Muhammad Babar Javed, William Shotyk</u> [47] <i>Concentrations of heavy metals (Ag, As, Cd, Pb, Sb, Tl) in Athabasca bituminous sands are comparable to crustal values</i>	4:20	<u>Jun Han, Karen Lin, Yi Yi, John J. Gibson, Jean Birks, Christoph H. Borchers</u> [5] <i>Qualitative and quantitative profiling of naphthenic acids by polar reversed-phase liquid chromatography - high-resolution mass spectrometry</i>
<u>Mark Donner, William Shotyk, Kevin Francesconi, Tariq Siddique</u> [37] <i>Arsenic and selenium in the lower Athabasca River, Alberta</i>	4:40	<u>Sheher Bano Mohsin</u> [21] <i>GC-APCI coupled with Ion Mobility High Resolution Mass Spectrometry for the Analysis of Sulfur Compounds in Fuel</i>
<u>Jon Fennell</u> [40] <i>Thermal mobilization and Arsenic: implications for the oil sands industry</i>	5:00	
<b>6:00 – 10:00 Banquet and Karasek Awards Sponsored by Wellington Laboratories KC 105</b>		

## Tuesday Afternoon, July 14

### Dedicated Poster Session

2:40-3:40 PM

KC 203

Posters must be installed no sooner than 10 a.m. Monday morning, July 13, and must be ready for viewing by 1:20 p.m. Monday. Posters must be removed by 4 p.m. Tuesday, July 14. Authors must stand by their posters during the 2:40 p.m. to 3:40 p.m. Poster/Exhibitor viewing period on Tuesday. The best student poster will be eligible to win the Karasek Award. Please bring your own fasteners. Posters left in the ballroom after 4:00 PM Tuesday will be discarded! Poster abstracts appear at the back of this program.

Authors	Title
<u>Yu Chieh Wang</u> , Youn Yuen Shu, JiaLin Wang, Jen-Fon Jen	<i>Optimization of Dispersive Liquid–Liquid Microextraction Coupled with Gas Chromatography-Negative Ion Chemical Ionization Mass Spectrometry for the Determination of Pyrethroids in Agricultural Products</i> [15]
<u>Aaron Hineman</u> , Chady Stephan	<i>Effect of Dwell Time on SP-ICP-MS Data Acquisition Quality</i> [20]
<u>Elham Zeinijahromi</u> , Sylvia Tiu, Lorinda Butlin, David W. Kinniburgh	<i>Effect of Storage Temperature and Holding Time on the Stability of Inorganic Anions in Drinking Water Samples</i> [23]
<u>Youn Yuen Shu</u> , Yu Chieh Wang, Yun Yun Sun, Robert C Lao, Jen-Fon Jen	<i>Purge-Assisted and Temperature Controlled Headspace Solid-Phase Microextraction for the Determination of Phthalate Esters in Water Samples</i> [25]
<u>Narumol Jariyasopit</u> , Yongchun Liu, John Liggio, Tom Harner	<i>Stability of Polycyclic Aromatic Compounds in Polyurethane Foam-type Passive Air Samplers upon O<sub>3</sub> Exposure</i> [31]
<u>Beatriz Araujo</u> , Brian Dimock, Rodrigo Lima Sobrinho, Marcelo Bernardes, Marcelo Almeida, Carlos Eduardo Rezende	<i>Seasonal Variability of Total Mercury and Methylmercury in the Amazon floodplain lakes –Brazil</i> [33]
<u>Jen-Fon Jen</u> , Vinoth Kumar Ponnusamy	<i>A Novel Multiwall-Carbon Nanotubes Immobilized Hollow-Fiber Membrane Based Liquid Phase Microextraction for the Analysis of Paclobutrazol in Aqueous Samples by Liquid Chromatography</i> [46]

## Wednesday Morning, July 15

### Metals in the Environment KC 201

Session Chair: Graeme Spiers

### Water KC 205

Session Chair: Ray Clement

<u>William Shotyk</u> [53] <i>Heavy metals in the Athabasca River: do the bituminous sands have a silver lining?</i>	8:40	
<u>Beatriz Araujo</u> , <u>Brian Dimock</u> , <u>Marcelo Almeida</u> , <u>Ana Paula Falcão</u> , <u>Carlos Eduardo Rezende</u> [32] <i>Methylmercury in sediments from shelf and continental slope at Campos Basin near Rio de Janeiro, Brazil</i>	9:00	<u>Susan Richardson</u> [8] <i>The Next Generation of Drinking Water Disinfection By-Products: Occurrence, Formation, Toxicity, and New Links with Human Epidemiology</i>
<u>Muhammad Babar Javed</u> , <u>Tariq Siddique</u> [48] <i>An arsenic speciation method optimized for arsenite, arsenate and DMAA species in river water and sediment using IC-ICP-MS</i>	9:20	<u>Vasile Furdui</u> , <u>Stefanie Maedler</u> , <u>Teresa Switzer</u> , <u>Fengrong Sun</u> , <u>Cindy Tat</u> , <u>Robert Tooley</u> , <u>Matt Pamuku</u> , <u>H. M. Skip Kingston</u> [52] <i>Trace and ultra-trace analysis of chromium (VI) by ion chromatography tandem mass spectrometry</i>
<u>Rathika Balthasar</u> , <u>Carola Sanpera</u> , <u>Sergi Díez</u> [27] <i>Use of Mercury stable isotopes in bird feathers to identify spatial and dietary trends of mercury pollution</i>	9:40	<u>Holger Hintelmann</u> [43] <i>Fate of silver nanoparticles in lake water</i>
<u>Ruoyu Sun</u> [26] <i>The preconcentration of ultra-trace mercury from coral skeleton by a double-stage tube furnace—acid-trapping protocol for total mercury and stable mercury isotope ratio measurement</i>	10:00	<u>Jean-Luc Brousseau</u> , <u>Zoe Grosser</u> , <u>William Jones</u> , <u>David Gallagher</u> , <u>Michael Ebitson</u> [50] <i>Rugged Method 625 for Wastewater Extraction by Solid Phase Materials</i>
10:20 Refreshments KC 2 <sup>nd</sup> Floor Galleria		
<p>11:00 – Plenary Sponsored by LECO KC 201</p> <p><u>Susan Richardson</u></p> <p><i>Safe Drinking Water? Effect of Wastewater Inputs and Source Water Impairment and Implications for Water Reuse</i></p> <p>(Abstract 7)</p>		
EnviroAnalysis 2015 Close		

## **PLATFORM PRESENTATIONS ABSTRACTS**

### **Abstract 1**

#### **The Direct Analysis of Multiple Elements in 25% Brines using an Agilent 7900 ICP-MS with an Ultra High Matrix Interface**

Pamela Wee

Agilent Technologies Canada Ltd, Mississauga, Ontario, Canada

The analysis of solutions containing high total dissolved solids (TDS) by ICP-MS can be challenging. Problems from clogging of the interface cones, poor run stability, and non-spectral interferences, are often mitigated by dilution. A suitable diluent can be difficult to procure, either due to its inadequate purity or the insolubility of some analytes. The use of an Ultra High Matrix Interface (UHMI) allows sample aerosols to be diluted by high purity argon during transfer into the torch.

The effectiveness of the Agilent 7900 ICP-MS with UHMI in directly analyzing multiple elements in 25% brines will be discussed. An octopole collision reaction cell was used for optimal spectral resolution and detection capability. Pure helium was used for the resolution of polyatomic interferences on several elements, such as  $^{40}\text{Ar}^{35}\text{Cl}^+$  and  $^{40}\text{Ca}^{35}\text{Cl}^+$  on mono-isotopic  $^{75}\text{As}^+$ . Pure  $\text{H}_2$  was used to chemically resolve interferences on some elements such as  $^{40}\text{Ar}^{38}\text{Ar}^+$  and  $^{40}\text{Ca}^{38}\text{Ar}^+$  on  $^{78}\text{Se}^+$ . Basic concepts and many examples will be presented.

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### **Abstract 2**

#### **Implications of Performance Metrics on the Quality of ICP-MS Analysis**

Pamela Wee

Agilent Technologies Canada Ltd, Mississauga, Ontario, Canada

Despite popular belief, attaining the highest possible sensitivities in ICP-MS analysis does not always result in the lowest detection limits, nor does it provide optimal analytical conditions for all applications. Unless the sample matrix is a simple one, or removed prior to introduction to the MS, highest sensitivities can result in increased background, more noise, and poor long term run stability. This leads to increased frequency of QC failures, maintenance, and instrument downtime. Hence adequate sensitivities that deliver the required method detection limits (MDL) should be the ultimate practical goal.

Another commonly used metric is the cerium oxide to cerium ( $\text{CeO}^+/\text{Ce}^+$ ) ratio. This ratio serves as an indicator of polyatomic ion interferences, such as  $^{35}\text{Cl}^{16}\text{O}^+$  on  $^{51}\text{V}^+$ . The lower the  $\text{CeO}^+/\text{Ce}^+$  ratio, the less severe these interferences. But to attain this, sensitivities will be reduced and may deteriorate MDLs. In some ICP-MS systems, a decrease in the  $\text{CeO}^+/\text{Ce}^+$  ratio is accompanied by a significant increase in the

Ce<sup>2+</sup>/Ce<sup>+</sup> or Ba<sup>2+</sup>/Ba<sup>+</sup> ratio. The higher this ratio, the worse the severity of doubly-charged ion interferences, such as <sup>150</sup>Nd<sup>2+</sup> or <sup>150</sup>Sm<sup>2+</sup> on <sup>75</sup>As<sup>+</sup>. Since polyatomic interferences are more common than doubly-charged ion interferences, the Ba<sup>2+</sup>/Ba<sup>+</sup> ratio is often sacrificed in order to minimize the CeO<sup>+</sup>/Ce<sup>+</sup> ratio.

This paper investigates the effects of sensitivity and Ba<sup>2+</sup>/Ba<sup>+</sup> ratio on instrument detection limits and spectral interferences, with and without the use of various gases in a collision reaction cell.

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### Abstract 3

#### Determination of Total Mercury in Fish Muscle Samples by Inductively Coupled Plasma Mass Spectrometry

Xinbang Feng, Lisa Shi, Julius Pretorius  
Alberta Innovates Technology Futures, Vegreville, Alberta, Canada

A closed vessel microwave digestion method was developed for dissolution of fresh fish muscle samples, followed by total mercury determination using ICP-MS. Fresh fish muscle samples were digested with nitric and hydrochloric acid under microwave heating for total mercury determination. Memory effect of mercury in ICP-MS was removed by using a high throughput flow injection ESI-SC FAST autosampler and adding a 100 ppb gold spike to each analytical tube including blank, wash, standards and samples. Analytical results of certified reference materials were 20 to 30% lower due to the matrix effect which suppressed the mercury signals in ICP-MS. The matrix effect was diminished by diluting the digested sample solutions up to 5-fold. Results obtained from ICP-MS analyses were compared with the results obtained from analyses conducted with Cold Vapour Atomic Fluorescence Spectrometry. Good agreement was observed.

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### Abstract 4

#### Microwave Digestion of Biological Tissue Standard Reference Materials for Multi-elemental Determination by ICP-MS

Lisa Shi, Xinbang Feng, Julius Pretorius  
Alberta Innovates Technology Futures, Vegreville, Alberta, Canada

A closed vessel microwave digestion method was developed for biological standard reference materials including plant and animal tissue matrices, followed by trace elemental determination using ICP-MS. Loss of volatile elements in the microwave digestion was minimal. There were three microwave digestion procedures performed in this experiment; nitric acid digestion only and mixtures of acid digestions including nitric acid and hydrogen peroxide, and nitric and hydrofluoric acid. The ICP-MS spectral characteristics produced from these digestion procedures were compared and contrasted. The ICP-MS instrument was operated in three modes – Standard mode, Collision mode and Reaction mode configurations. Percentage recoveries of metals from the standard reference materials were significantly improved since the potential spectral interferences from biological matrix components in ICP-MS spectra were suppressed by using dynamic reaction cell and kinetic energy discrimination techniques.

## Abstract 5

### Qualitative and quantitative profiling of naphthenic acids by polar reversed-phase liquid chromatography - high-resolution mass spectrometry

Jun Han<sup>1</sup>, Karen Lin<sup>1</sup>, Yi Yi<sup>2</sup>, John J. Gibson<sup>2</sup>, Jean Birks<sup>3</sup>, Christoph H. Borchers<sup>1,4</sup>

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<sup>2</sup> Alberta Innovates – Technology Futures, Victoria, British Columbia, Canada

<sup>3</sup> Alberta Innovates – Technology Futures, Calgary, Alberta, Canada

<sup>4</sup> Department of Biochemistry and Microbiology, University of Victoria, Victoria, British Columbia, Canada

Naphthenic acids (NAs) are a group of aliphatic and polycyclic organic acids naturally occurring in crude oils and bitumen. There have been a lot of concerns about the acute and chronic toxicities of NAs, especially the classic naphthenic acids (cNAs;  $C_nH_{2n+2}O_2$ ), to aquatic systems. In this work, we propose an improved method by polar reversed-phase ultrahigh-performance liquid chromatography coupled to high-resolution mass spectrometry (PRP-UPLC-HRMS) for qualitative and quantitative profiling of cNAs in both commercial NA standard substance mixtures and oil sands process water (OSPW).

Chromatographic separations of cNAs were compared on different reversed-phase UPLC columns. A polar CN-bonded phase generated narrow and symmetric chromatographic peaks for all the detected cNAs. Online Fourier transform MS indicated significant effects of the mass resolution on compound detection. Identification of cNAs was based on the accurately-measured molecular masses (errors  $\leq 2$  ppm), correlation of LC retention times with elemental compositions of cNA homologues, and structural confirmation of putative cNAs by COOH-specific chemical derivatization. As a result, this PRP-UPLC-HRMS method enabled successful assignments of more than 300 different molecular formulae of cNAs from the analyses of two commercial standard substance mixtures, and up to 448 molecular formulae corresponding to 630 putative cNA compounds from the analyses of two representative OSPW samples generated by two oil sands mining processes (hot water extraction and steam assistant gravity drainage). These results indicated a complicated cNA profile of OSPW than that of the commercially available standard mixtures, and the two OSPW samples showed significantly different cNA compositions and abundance distributions from the standard substances and from each other. Absolute quantitation of the cNAs with external calibration showed good linearity ( $R^2 = 0.9999$ ) and a lower on-column limit of quantitation (0.27  $\mu\text{g}$ ). The quantitation accuracy ranged from 65% to 80% (CV%  $\leq 4.5$ ), as measured from standard-substance spiking tests. In summary, this work allowed a more comprehensive detection of cNAs and revealed the most complicated profile of cNAs thus far.

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## Abstract 6

### An isotopic approach to source apportionment of PAHs in the Athabasca oil sands region

Jason Ahad<sup>1</sup>, Josué Jautzy<sup>2</sup>, Anna Smirnoff<sup>1</sup>, Charles Gobeil<sup>2</sup>, Martine Savard<sup>1</sup>

<sup>1</sup> Natural Resources Canada, Québec, Canada

<sup>2</sup> Institut national de la recherche scientifique, Québec, Canada

The continued expansion of oil sands mining and upgrading activities in Canada's Athabasca oil sands (AOS) region has led to concerns about emissions of contaminants such as polycyclic aromatic hydrocarbons (PAHs). PAHs, which are found at naturally high levels in petrogenic sources such as AOS bitumen, are also produced pyrogenically during the incomplete combustion of organic matter and biologically during early sediment diagenesis. Understanding the impact of oil sands mining activities on the surrounding environment thus requires techniques which can discriminate between these different sources. Here, we present results from several studies carried out under the framework of Natural Resources Canada's CORES Project (Coal & Oil sands Resources Environmental Sustainability, 2009-2014) which utilised compound-specific isotope analysis ( $\delta^{13}\text{C}$ ,  $\delta^2\text{H}$  and  $\Delta^{14}\text{C}$ ) to delineate and quantify sources of PAHs in the AOS region. Analyses were carried out in dated lake sediments to provide an important temporal component. Stable carbon isotope ( $\delta^{13}\text{C}$ ) characterisation suggested a fugitive dust source for PAHs deposited in a headwater lake situated 55 km southeast of the main area of mining operations, and for a predominantly wildfire contribution to PAHs deposited in northwest Saskatchewan lakes located between 100 to 220 km east-northeast of the AOS. The application of both  $\delta^{13}\text{C}$  and stable hydrogen isotope ( $\delta^2\text{H}$ ) analysis allowed for an improved delineation of potential mining-related inputs and provided evidence for oil sands petroleum coke (petcoke) in a small lake in the Peace-Athabasca Delta (PAD) situated around 150 km north of the center of mining operations. In a study examining the sources of background PAHs delivered to the PAD via the Peace River watershed, radiocarbon isotopes ( $\Delta^{14}\text{C}$ ) indicated a predominantly natural petrogenic source (93% petrogenic, 7% forest fire) for alkylated PAHs during the past ~50 years. This research has demonstrated that molecular level isotope measurements provide a powerful tool to identify and quantify natural and anthropogenic sources of PAHs in the AOS region.

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## Abstract 7

### Safe Drinking Water? Effect of Wastewater Inputs and Source Water Impairment and Implications for Water Reuse

Susan Richardson

University of South Carolina, Columbia, South Carolina, USA

The elimination of emerging contaminants (ECs) during conventional wastewater treatment is not complete, and therefore, they are continuously released via wastewater effluents into the aquatic environment. This constitutes a major issue for water reuse because these compounds can undergo transformation in the environment or during disinfection if reclaimed water is used for drinking water production. Different ECs, e.g., perfluorinated compounds, pharmaceuticals, antibacterials, plasticizers, and preservatives, and their transformation products, are sometimes found in finished drinking waters. This presentation will review ECs detected in drinking water and disinfection by-products (DBPs)

generated by different ECs present in the aquatic environment, as well as DBPs formed by other source water impairments brought about by climate change. Moreover, the potential toxicologic effects that these pollutants and their transformation products pose for human health will also be discussed, as well as advanced treatment technologies that might be used to remove them.

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## Abstract 8

### **The Next Generation of Drinking Water Disinfection By-Products: Occurrence, Formation, Toxicity, and New Links with Human Epidemiology**

Susan Richardson

University of South Carolina, Columbia, South Carolina, USA

Drinking water disinfection by-products (DBPs) are an unintended consequence of using disinfectants to kill harmful pathogens in drinking water. DBPs are formed by the reaction of disinfectants with natural organic matter (NOM) and bromide or iodide; precursors can also involve pollutants, such as pesticides, pharmaceuticals, antibacterial agents, estrogens, textile dyes, bisphenol A, parabens, surfactants, and algal toxins. In addition, DBPs can be formed in swimming pool water. Eleven DBPs are currently regulated in the United States, but more than 600 have been identified. Human health concerns include bladder cancer, miscarriage, and birth defects in drinking water and also asthma from heavy exposure to indoor swimming pools. Emerging, unregulated DBPs include halonitromethanes, iodo-trihalomethanes, iodo-acids, haloamides, halofuranones, haloacetonitriles, nitrosamines, and halobenzoquinones. Many of these unregulated DBPs are more genotoxic or cytotoxic than those currently regulated. This presentation discusses these issues, along with precursors and mechanisms for the formation of this next generation of DBPs, as well as new links with epidemiology.

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## Abstract 9

### **Tandem Mass Spectrometry and High Resolution Mass Spectrometry Uncovers Chlorination Reaction Pathway for X-Ray Contrast Media in Drinking Water Treatment**

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Iodinated X-ray contrast media (ICM) are widely used for medical imaging, at doses up to 200g per person. They are excreted intact in ~24 h, and are extremely stable during wastewater treatment, such that they are found at the highest levels (up to 100 ppb) of any pharmaceutical in the environment. Drinking water sources downstream can be impacted, with ppb levels of ICM measured in drinking water reservoirs. While ICM are non-toxic themselves, they can transform into the most toxic disinfection byproducts (DBPs) known. In this new research, we are using high resolution-MS and tandem-MS to uncover the chlorination reaction pathways for the transformation of these medical imaging compounds in drinking water treatment. We collected source waters from different geographic

locations (GA, NC, OH, SC, and Germany) and carried out controlled laboratory reactions with ICM and different disinfectants (chlorine, chloramines, and chlorine dioxide) at different pH conditions used in drinking water treatment. We used LC-high resolution-MS and LC-tandem-MS using a Thermo LTQ Orbitrap Velos mass spectrometer (60,000 resolution), and GC-high resolution-MS using a LECO HRT time-of-flight mass spectrometer (25,000 and 50,000 resolution) to identify both high molecular weight reaction intermediates and low molecular weight iodinated DBPs in order to determine reaction mechanisms. Preparative-LC was used to isolate high-molecular-weight DBPs for additional structural confirmation by NMR and for toxicity testing. Cytotoxicity and genotoxicity of the reaction mixtures and 5 isolated high-molecular-weight DBPs were measured.

High resolution-MS and MS-MS were key in providing molecular formula and structural information in the identification of the reaction products and determination of formation mechanisms. Iopamidol was by far the most reactive of the ICM compounds studied, with reactions occurring with both chlorine and chloramine, and very minimal or no reaction with iopromide, iohexol, iomeprol, or diatrizoate.

Overall, 19 high molecular weight DBPs were identified for reactions of iopamidol with chlorine, along with several low molecular weight DBPs, including iodo-acids, iodo-trihalomethanes, and three new iodo-DBPs not previously known. Reaction mechanisms involved an initial attack of chlorine on one of the amide side chains (side chain A) of iopamidol, exchange of chlorine for iodine, inversion of side chain A, amide hydrolysis, cleavage of side chain B, and oxidation of an amino group to a nitro group. Overall, chlorine was more reactive than monochloramine, which was more reactive than chlorine dioxide. In addition, while iopamidol reacts to form DBPs in the absence of natural organic matter (which is naturally present in drinking water sources), the presence of natural organic matter greatly enhances the formation of iodo-DBPs.

NMR spectroscopy was used to support the identifications of two DBPs, DBP705 and DBP778 (with molecular weights of 705 and 778 Da, respectively), through the analysis of these isolated DBPs from the reaction mixtures. Four of the 5 isolated DBPs that were tested for cytotoxicity (DBP551, DBP735, DBP643, and DBP705) were found to have measurable levels of cytotoxicity, but at much lower levels compared to the low molecular weight iodo-DBPs, such as iodoacetic acid. Cytotoxicity and genotoxicity of the reaction mixtures consistently show that source waters containing iopamidol show much higher toxicity after being reacted with chlorine than waters not containing iopamidol.

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## **Abstract 11**

### **Inexpensive Rapid Method for Pops Analysis of Food Using ASE and Tandem Acid Silica/Carbon Columns**

Dave Hope

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#### **Introduction**

Conventional methods for PCDD/F and dI PCB analysis in food have been laborious and expensive, using soxhlet extraction, multi-column clean-ups (acid/base silica gel, Florisil, alumina, carbon) and ultimately high resolution mass spectrometry for analysis. Automated alternatives have been posed for the extraction and clean-up, however the costs can be prohibitive for smaller labs, especially in a competitive marketplace.

Lee et al<sup>1</sup>, and Yang et al<sup>2</sup> have demonstrated the use of tandem acid silica / carbon columns in analyzing environmental samples for PCDD/F and dI PCB. We have taken previously analyzed food samples and developed a method for extraction by Accelerated Solvent Extraction (ASE) using Dionium™ cells. Samples containing up to 5 g of lipid are cleaned up on acid silica gel columns and by coupling with a carbon column (Cape Technologies), can separate PCBs from PCDD/F.

### Materials and methods

Cleanliness and potential for carry-over on the ASE was evaluated using blanks and fortified samples. Cells were packed with fortified samples containing 1-2 ng of PCDD/F and 1-5 ng of 72 PCB congeners.

Analyte fractionation was determined using standards containing all 209 PCB congeners and seventeen 2,3,7,8-PCDD/F congeners. While many PCBs were eluted with 30 mL hexane, it requires a further 6 mL of 1:1 toluene/hexane to elute the 12 dI PCBs. All PCDD/F are eluted by reversing the flow on the carbon column and eluting 30 mL of toluene.

Unfortified samples (butter, horse fat, CARP2) were spiked with 0.5-1.0 ng of fifteen <sup>13</sup>C-PCDD/F standards, and twelve <sup>13</sup>C-dioxin-like PCB standards, ground with 10 g of diatomaceous earth and then packed into a 100 mL Dionium™ cell as follows: 30 mm cellulose filter, 10 g Dioxex ASE Prep CR Na+ form (sulphonated divinyl benzene/styrene copolymer), sample and top with diatomaceous earth to 10 mm from top of cell. The ASE was run under the following conditions: Extraction Solvent: Hexane; Temperature: 100 °C; Pressure: 1600 psi; Heat Time: 5 min; Static Time: 5 min; Flush Volume: 70%; Purge Time: 120 s; Static Cycles: 3; Total Extraction Time: 25 min per sample. Extracted samples (1-5 g lipid) were rotary evaporated to 10 mL. CAPE Technologies immunoassay tandem acid silica / carbon column sample preparation kits were used for sample clean-up. Carbon columns were prerinsed with solvent in the following order: toluene, dichloromethane and hexane, and then coupled to a prerinsed (hexane) acid silica column. Samples were applied to the acid silica gel column and the system was pressurized to 10 psi with nitrogen to give a flow rate of 1 mL per minute. Thirty mL of hexane was eluted through the tandem column system (F1). The carbon column was removed from the acid silica column and placed on a clean empty glass column. Six mL of 1:1 toluene/hexane was eluted (F2) containing all 12 dI PCBs (see Figure 1).

Lastly, the direction of flow was reversed through the carbon column by inverting it. Thirty mL of toluene was passed through the column and collected (F3) containing all PCDD/F. F2 and F3 were concentrated to near dryness and fortified with injection standards prior to analysis by GC/HRMS. All analyses were performed on a Trace GC coupled to a Thermo Scientific DFS HRMS following protocols set out in EPA 1613b and 1668C.

### Results and discussion

Using the clean-up we were able to separate the PCBs from PCDD/F. Further, we were able to isolate the 12 dI PCBs from potential coeluting PCBs of high level of chlorination (e.g. PCB110 and PCB203 elute in F1 and therefore cannot interfere with PCB81 and PCB169 respectively). A total of 61 PCBs elute in F2, leaving 148 in F1. Unfortunately, not all the marker PCBs are in F1 (PCB28 is in F2). It is interesting to note that the cut off points in each level of chlorination follow the elution order found on an SGE HT8 column (i.e. all tetrachloro congeners eluting on an SGE HT8 column at or before PCB040 are in F1, while those eluting after are found in F2). The first PCB to elute in F2 is: DiCB9, TriCB23, TetraCB57, PentaCB124, HexaCB167 and HeptaCB189). MonoCBs elute in F2, while OcCB, NoCB and DeCB elute in F1.

Ten samples of CARP2 were analyzed for PCDD/F using ASE and Cape columnning. NRC lists the lipid content to be approximately 7%. Results on a fresh weight basis (see Figure 2) fall within the range of certification. RSDs were below 20% except when concentrations fell below 1.3 ng/kg. High lipid food samples (butter and horse fat) were extracted with the ASE and subsequently columnned on tandem acid silica / carbon columns (analyzed in replicate, n=5). Previously these samples were extracted using our conventional methodology (dissolving in 3N acid, back extraction with 9:1 dichloromethane/acetone and clean-up on acid/base silica gel followed by basic alumina (control sample). Data for horse meat is presented in Figure 3. The average PCDD/F values for horsemeat matches very well with the Control sample. The relative standard deviation (RSD) for five replicates increases as the concentration decreases. The PentaCB values are lower for the samples analyzed by ASE, while the others are comparable. As the Control sample is only a single data point, it is possible that there was an error in its original analysis. <sup>13</sup>C-labelled standard recoveries were acceptable for all samples. The average PCDD/F recovery was 81% with an RSD of 15%, while the PCBs recovered at 51% with an RSD of 14%.

Butter samples were analyzed in a manner similar to horse meat. In addition, five butter samples were fortified with native PCDD/F and PCBs, then analyzed. The results are presented in Figure 4. The butter was at background concentrations. <sup>13</sup>C-standard recoveries for PCDD/F ranged from 23-78% with an average of 42%.

In conclusion, ASE combined with Cape Technologies columns provides an acceptable method for analyzing food samples, including those with high lipid concentrations. ASE provides a rapid extraction of <30 minutes per sample. The Cape columns allow for the separation of PCBs from PCDD/F and provide an inexpensive clean-up procedure, with consumable costs of <\$20 per sample.

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## Abstract 12

### Evaluation of TSQ8000 (GC-MS/MS) for PCDD/F Analysis

Dave Hope

Pacific Rim Laboratories Inc., Surrey, British Columbia, Canada

#### Introduction

In 2012, the European Food Safety Authority announced that it would accept PCDD/F (polychlorinated dioxin and furan) data for food and feed products generated by gas chromatography coupled with a triple quadrupole mass spectrometer (GC/MS/MS). Previously, only data generated by high resolution mass spectrometry (GC/HRMS) had been deemed acceptable<sup>1,2,3,4</sup>. The change in legislation is expected to be complete in mid-2014. This paper will describe the evaluation of a Thermo Scientific TSQ8000 (GC-MS/MS) for use with food extracts. A comparison study was performed on quality control samples and food samples of animal origin to evaluate the robustness of a new extraction and clean-up procedure followed by analysis on a TSQ8000. All results demonstrate the suitability of the extraction/clean-up and subsequent GC-MS/MS analysis.

#### Materials and methods

Samples: CARP2, ground whole carp reference material, National Research Council Canada

Reagents: Native and <sup>13</sup>C-labelled PCDD/F standards were purchased from Wellington Labs. Canada. Solvents used were Pesticide Grade from Caledon Laboratories, Canada.

Apparatus

GC-HRMS: Thermo Scientific Trace GC, DFS HRMS

GC-MS/MS: Thermo Scientific Trace 1310 GC, TSQ8000 MS/MS

RX-DIOXIN2 column, 60 m x 0.25 mm i.d., 0.25 µm film thickness

ASE 350 – accelerated solvent extraction

SRM CARP2 samples were fortified with <sup>13</sup>C-labelled PCDD/F standards and extracted using ASE followed by clean-up on tandem acid silica / carbon columns. The samples were concentrated to 20 µL and analyzed by GC/HRMS and subsequently by GC-MS/MS on a TSQ8000. For MS/MS analysis, two ions from the M-COCl cluster were monitored. All injections were made with a split/splitless injector.

### Results and discussion

The TSQ8000 showed excellent linearity with PCDD/F standards. A calibration curve ranging from 0.02-200 pg/ µL for TCDD/F (5x higher for PeCDD/F, HxCDD/F and HpCDD/F; 10x higher for OCDD/F) was obtained with RSDs averaging at 9% and ranging from 3.4-12.4%. The instrument has little noise therefore it is important to be able to have the lowest calibration point possible as this will determine your Limit of Quantitation. For a 10 g sample concentrated to 20 µL, a 0.02 ng/ µL calibration point will give an LOQ of 0.04 ng/kg.

Ten samples of the SRM CARP2 were analyzed by both HRMS and MS/MS. The reproducibility (as measured by the average relative standard deviation) for nine certified PCDD/F congeners was 12.2% for HRMS and 10.7% for the MS/MS, with ranges of 5-32% and 6-14% respectively. The TEQ for the same 9 congeners was 15.3 ng/kg (HRMS) and 16.1 ng/kg (MS/MS), with an expected range from 13.3-17.8 ng/kg.

Another advantage of MS/MS technology is that by monitoring the M-COCl cluster, major HRMS interferences like chlorinated diphenylethers are no longer a problem as they cannot produce an M-COCl ion. The TSQ8000 GC-MS/MS has the sensitivity and reproducibility to produce quality PCDD/F data.

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### Abstract 13

#### Anatomy of an Oil Spill and its Subsequent Chemical Evolution by Ultrahigh-Resolution Fourier Transform Ion Cyclotron Resonance Mass Spectrometry

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Ultrahigh resolution FT-ICR mass spectrometry and comprehensive two-dimensional GCxGC analysis enable comprehensive molecular level characterization of the 2010 Macondo Deepwater Horizon crude oil released into the Gulf of Mexico. We have generated a library archive of the wellhead crude oil, as well as tar balls washed ashore. GCxGC accesses volatile, non-polar, low-molecular weight constituents, whereas FT-ICR MS assesses polar, involatile, high molecular weight species. The combination provides unprecedented insight into environmental evolution of oil spills: e.g., composition-specific chemical analysis, and molecular discrimination between biotic and abiotic weathering for current and future oil spills. Work supported by NSF DMR-11-57490, the BP/The Gulf of Mexico Research Initiative to the Deep-C Consortium, the Florida State University Future Fuels Institute, and the State of Florida.

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## Abstract 14

### On-line solid-phase extraction coupled to liquid chromatography tandem mass spectrometry for the analysis of cyanotoxins in algal blooms

Paul Fayad<sup>1</sup>, Audrey Roy-Lachapelle<sup>1</sup>, Sung Vo Duy<sup>1</sup>, Michele Prevost<sup>2</sup>, Sebastien Sauve<sup>3</sup>

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<sup>3</sup>Université de Montréal, Québec, Canada

An analytical method based on on-line SPE-LC-HESI-MS/MS has been developed for the detection and quantification of eight selected cyanotoxins in algal bloom waters that include microcystins, anatoxin-a and cylindrospermopsin. The injection volume was 2 mL according to the expected concentration of cyanotoxins in matrix. The method shows an analysis time of 7 min per sample, acceptable recovery values (91-101 %), good precision (RSD < 13 %) and method limits of detection at the low-microgram per liter levels (0.01-0.02 µg L<sup>-1</sup>). In particular, a detailed discussion on optimization parameters that have an impact on the overall performance of the method has been presented. In addition, method development permitted the chromatographic separation of anatoxin-a and phenylalanine, an isobaric interference with a similar chromatogram. All optimization and validation experiments for the on-line SPE method and chromatographic separation were performed in environmentally relevant algal bloom water matrices. The applicability of the method was tested on several algal bloom water samples from monitored lakes across the province of Québec (Québec, Canada) known to produce cyanotoxins. All of the studied cyanotoxins were detected with the exception of cylindrospermopsin. In addition, microcystin-LR concentrations (between 1.2 and 36 µg L<sup>-1</sup>) in several samples exceeded the proposed guidelines from the World Health Organization of 1 µg L<sup>-1</sup> in drinking water for both free and cell-bound microcystin-LR.

## Abstract 17

### **A non-targeted chemical analysis strategy for identification of dissolved organic chemicals that cause acute toxicity of oil sands process-affected water**

Hui Peng, Hattan Harbi, Jianxian Sun, Steve Wiseman, Paul Jones, John P. Giesy  
University of Saskatchewan, Saskatchewan, Canada

Previous studies have shown that oil sands process-affected water (OSPW) can cause multiple acute and chronic toxicities, however, it is difficult to identify the causative agents due to complexity of OSPW. This study established a novel strategy that combined untargeted chemical analysis and biochemical fractionation, to robustly identify chemicals in relatively fresh OSPW from Base Mine Lake (BML-OSPW) that were causing toxicity. The strategy was used to identify chemicals causing toxicities with or without known protein targets. For example in acute toxicity for which protein targets are unknown, a solid phase extraction method was developed for fractionation of the dissolved organic chemicals in BML-OSPW and it was found that acute toxicity was specifically detected in the DCM/Hex fraction, which showed 5-10 fold greater toxicity than other fractions of BML-OSPW or aged OSPW from the Pond 9 experimental reclamation pond. A computation algorithm was used in untargeted detection of compound peaks in samples, and 155,892 and 80,830 peaks were detected by use of HPLC-Orbitrap ultra-high resolution mass spectrometry in positive and negative mode, respectively. Peaks that were of greater abundance in toxic fractions than nontoxic fractions were expected to be potential causative compounds. After constraining the multiple cutoff values according to toxicity results, the number of putative causative compounds was narrowed to 83. Molecular formulae were calculated for the 83 compounds based on isotope distribution and exact mass. Chemical structures of some compounds were searched from in silica databases by combining MetFrag and MetFusion software. Results of this study demonstrate that a combination of untargeted chemical analysis and biochemical fractionation can be used to efficiently identify chemicals in complex mixtures of OSPW that caused specific types of toxic effects.

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## Abstract 19

### **Single Particle ICP-MS (SP-ICP-MS) for the Detection of Metal-Based Nanoparticles in Environmental Matrices**

Aaron Hineman, Chady Stephan  
PerkinElmer Canada, Woodbridge, Ontario, Canada

The use of nanoparticles in consumer products is showing a tremendous increase over time. The National Institute of Standards and Technologies reported that nanotechnology-based consumer products are currently entering the market at rate of 3 to 4 per week, and it is estimated that \$2.6 trillion in manufactured goods will contain nanotechnology by 2014[1].

In spite of their beneficial properties, possible risks for humans and the environment need to be thoroughly investigated, and multiple key characteristics need to be assessed. Detailed information about the quantity, shape, size, size distribution, structure, composition, surface charge and functionality need to be studied and addressed in order to perform an appropriate and reliable risk assessment.

This work presents Single Particle Inductively Coupled Plasma Mass Spectrometry (SP-ICP-MS) as a tool in assessing the fate of engineered nanoparticles in environmental sample types [2,3]. The technique allows for the differentiation between ionic and particulate signals, measurement of particle sizes and size distribution, and assists in monitoring agglomeration.

[1] National Institute of Standards and Technologies (NIST), Environmental Leaching of Nanoparticles from Consumer Products <http://www.nist.gov/mml/csd/inorganic/leachnano.cfm> (accessed October 11, 2013).

[2] Assessing the Fate of Silver Nanoparticles in Surface Water using Single Particle ICP-MS <http://www.perkinelmer.com/>

[3] Quantitative Evaluation of Nanoparticle Dissolution Kinetics using Single Particle ICP-MS: A Case Study with Silver Nanoparticles <http://www.perkinelmer.com>

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## Abstract 21

### GC-APCI coupled with Ion Mobility High Resolution Mass Spectrometry for the Analysis of Sulfur Compounds in Fuel

Sheher Bano Mohsin

Agilent, Schaumburg, Illinois, USA

This presentation discusses the use of ion mobility and high-resolution GC/MS for profiling sulfur compounds in a very complex sample such as diesel.

The economic value of diesel depends on its chemical composition. Molecules containing heteroatoms such as sulfur lower the value of fuel as they burn in air to produce smog. The amount of sulfur in diesel is being driven to lower levels because of environmental regulations.

The distribution of sulfur-containing compounds in various feedstocks is important as the refining process can be modified to meet regulations. Refineries use a process called hydrodesulfurization to remove sulfur from sulfur-containing compounds. For hydrodesulfurization, sulfur bound in aromatic structures such as benzothiophenes and dibenzothiophenes and their alkyl substituted analogs is the most difficult to remove catalytically. Therefore, it is important to study these species.

The profiling of sulfur compounds in diesel can be challenging because of the very complex matrix. There are thousands of different compounds in diesel, ranging from alkanes to alkenes to aromatics to heteroatomic sulfur, nitrogen, and oxygen compounds. The probability of completely resolving a single class of compound from all others in the sample is low.

Gas chromatography/high-resolution mass spectrometry is a useful tool when identifying peaks but, in the case of diesel, such attempts are usually frustrated by isobaric coeluting hydrocarbons. Ion mobility, coupled to GC/MS, provides an extra dimension of separation based on size, charge, and shape. After chromatographic separation, the ions formed in the source are separated again in gas phase in the

mobility cell. Larger ions traverse the drift tube at a slower rate than smaller ions resulting in gas-phase ion separation.

In this talk, we demonstrate the use of ion mobility for separating aromatic sulfur-containing compounds from the matrix. Two samples of diesel are compared – before and after hydrodesulfurization.

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## Abstract 22

### **Rapid And Sensitive Method For The Determination Of Polycyclic Aromatic Hydrocarbons In Soils Using Pseudo Multiple Reaction Monitoring Gas Chromatography/Tandem Mass Spectrometry**

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<sup>4</sup>Simon Fraser University, Burnaby, British Columbia, Canada

A method for the rapid determination of 18 polycyclic aromatic hydrocarbons (PAHs) in soil has been established based on a simplified solvent extraction and GC/MS/MS operated in pseudo multiple reaction monitoring mode (pMRM), a technique where the two quadrupoles mass monitor the same m/z. The pMRM approach proved superior to the classic single quadrupole technique, with enhanced sensitivity, specificity, and significant reduction in time consuming sample clean-up procedures. Trace level PAHs could be readily confirmed by their retention times and characteristic ions. The limit of quantitation in soil was observed to be 20 ng/g for 16 EPA-priority PAHs and 2 additional PAHs specific to Environment Canada. The developed method was linear over the calibration range 20–4000 ng/g in soil, with observed coefficients of determination of >0.996. Individual PAH recoveries from fortified soil were in the range 58.1 to 110.1%, with a precision between 0.3 and 4.9% RSD. The ruggedness of the method was demonstrated by the success of an inter-lab proficiency test study organized by the Canadian Association for Laboratory Accreditation. The present method was found to be applicable as a rapid, routine screening for PAH contamination in soil, with significant savings in terms of preparation time and solvent usage.

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## Abstract 24

### **Differentiating Natural and Anthropogenic Sources of Bitumen–Derived Organic Compounds to Water Resources Surrounding the Canadian Oil Sands Industry**

Jonathan Martin, Chenxing (Angela) Sun  
University of Alberta, Edmonton, Alberta, Canada

During oil sands surface mining in Canada, large volumes of water are used for the extraction of valuable bitumen, resulting in toxic oil sands process-affected water (OSPW) containing a complex mixture of bitumen-derived polar organic compounds. The OSPW is stored in large tailing ponds, some of which are close to the Athabasca River and its tributaries, raising concerns over potential seepage.

Nevertheless, natural discharge of groundwater that has been in contact with bitumen contributes a similar profile of polar organics to the aquatic system. Therefore, the objective of this study was to develop a forensic method that can be used to characterize the range of profiles in natural groundwater while also differentiating these from tailings pond seepage. A large set of groundwater, surface water and OSPW samples were collected from the oil sands development area in 2014. On-line solid phase extraction coupled with liquid chromatography / ultrahigh resolution Orbitrap mass spectrometry (on-line SPE-LC/Orbitrap MS) was developed for the characterization of thousands of organic species in these water samples. The on-line SPE-LC/Orbitrap MS method eliminates liquid-liquid extraction and minimizes sample preparation contamination, and also is highly sensitive, selective, and reproducible. All the organic compounds from each water sample analyzed by SPE-LC/Orbitrap MS under both positive and negative APCI were binned into heteroatom classes based on elemental compositions. Principal component analysis was carried out to compare the compositional differences between samples, thereby achieving source discrimination of bitumen-derived organic compounds in these samples.

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## Abstract 26

### **The preconcentration of ultra-trace mercury from coral skeleton by a double-stage tube furnace—acid-trapping protocol for total mercury and stable mercury isotope ratio measurement**

Ruoyu Sun

Trent University, Peterborough, Ontario, Canada

Annually-banded scleractinian corals are potentially a powerful registry of the chronological variation of trace elements in the ocean. However, accurate measurement of ultra-trace mercury concentrations in the coral aragonite ( $\text{CaCO}_3$ ) skeleton is very challenging (generally,  $[\text{Hg}]/[\text{Ca}] < 1 \text{ nmol/mol}$ , or  $< 2 \text{ ng Hg per g CaCO}_3$ ). Here, we use a double-stage tube furnace acid-trapping protocol to preconcentrate mercury into an oxidizing solution (2:1  $\text{HNO}_3/\text{HCl}$ , 40%, v/v). The mercury trapping solution is characterized by low procedural blanks, high recoveries and limited sample matrix transfer, and can be reliably analyzed for mercury concentrations by CV-AFS, and for stable mercury isotope ratios by MC-ICP/MS.

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## Abstract 27

### **Use of Mercury stable isotopes in bird feathers to identify spatial and dietary trends of mercury pollution**

Rathika Balthasar<sup>1</sup>, Carola Sanpera<sup>2</sup>, Sergi Díez<sup>3</sup>

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<sup>2</sup>Universitat de Barcelona, Spain

<sup>3</sup>Institute of Environmental Assessment and Water Research (IDÆA), Spanish National Research Council (CSIC), Madrid, Spain

Mass fractionation patterns of mercury stable isotopes can be used to identify mercury pollution from different food sources or areas of contamination. Precise measurement of mercury isotope ratios is possible through the use of Multi-collector Inductively Coupled Plasma Mass Spectrometry (MC-ICP-

MS). We have observed both Hg mass-dependent fractionation (MDF) and mass-independent fractionation (MIF) in a wide variety of materials and have found that bird feathers, in particular, show distinct isotope ratio differences based on nesting site proximity to mercury sources. Birds from marine sites had more positive  $\Delta^{199}\text{Hg}$  values (showing greater fractionation) than those from freshwater sites. Using this case study, we can test whether isotope ratios of Hg at the top level of the aquatic food chain (ie. piscivorous birds) can be used to successfully distinguish the Hg originating from a contaminated site (a chlor-alkali plant) from other Hg dietary sources (ocean water).

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## Abstract 28

### Determination of Volatile Organic Compounds in Ambient Air by Gas Chromatography Mass Spectrometry

Haiyan Helena Wang, Graham Knox, Julius Pretorius  
Alberta Innovates Technology Futures, Vegreville, Alberta, Canada

Volatile Organic Compounds (VOCs) in ambient air are analyzed by Gas Chromatography-Mass Spectrometry (GC-MS). Depending on the standard mixtures employed, up to 102 VOCs may be quantified. Standard mixtures employed include compounds from the US EPA's Ozone precursor mixture/ Photochemical Assessment Monitoring Stations (PAMS) and TO-15 compound lists. The analysis system consists of an Agilent GC-MS connected to a canister autosampler (Entech 7016D) and preconcentrator (Entech 7200). Air samples pass through the preconcentrator where moisture is removed using extended cold trap dehydration. Following dehydration, the sample components are focused by adsorption on a Tenax trap, and then injected into the GC-MS. The mass spectrometer is operated in combined SCAN and SIM modes. In SCAN mode, a wide range of mass to charge ratios is scanned continuously while, in SIM mode, only compounds from the target list are monitored. Target compound quantification is performed from SIM spectra using relative retention time and - abundances of two or more characteristic ions. Quantitation is accomplished by an internal standard technique using areas of characteristic ions. Tentatively Identified Compounds (TIC's) and their approximate concentrations are determined from data collected in SCAN mode. TIC's are identified from mass spectral libraries and detailed interpretation of the GC-MS data. The approximate concentration ranges of TIC's are determined using total ion profiles and response factors of the closest internal standards.

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## Abstract 29

### Quantification of Naphthenic Acids in Water Samples: Challenges for complex matrix?

Alberto dos Santos Pereira, James Srorey, Graham Knox, Julius Pretorius  
Alberta Innovates Technology Futures, Vegreville, Alberta, Canada

In Northern Alberta, Canada, the production of bitumen from open-pit oil sands mining requires 2-3 barrels of water for every barrel of bitumen produced. Large volumes of oil sands process-affected water (OSPW) are stored in large tailings ponds so that it can be recycled into the extraction process. Despite the water recycling efforts (80 to 95%), in 2012 the use of fresh water was approximately 187 million  $\text{m}^3$  (*Canadian Association of Petroleum producers 2013*); which is about 40% of the City of Toronto's annual water consumption. Moreover, due to the large water volume used daily, tailings

ponds are growing in volume and number, and in 2011 covered approximately 170 km<sup>2</sup>. There are concerns that OSPW leaches from tailings ponds into groundwater, or into river water, but proving this is complicated by the fact that natural groundwater can contain many of the same chemicals as OSPW (e.g. Naphthenic acids). As a consequence, there is an ongoing need to improve environmental monitoring in the Athabasca region.

A variety of techniques for the analysis of naphthenic acids have been proposed; ranging from simple fourier transform infrared (FTIR) to more sophisticated fourier transform ion cyclotron resonance (FTICR) mass spectrometry. In Alberta, gas chromatography coupled to mass spectrometry (GC-MS) is predominantly used by water monitoring agencies for naphthenic acid analysis. The main advantages of GC-MS are ease of operation and relatively low cost. However, it has been acknowledged that due to mass resolution limitations the results are subject to possible interferences from other compounds from the sample.

Here, we discuss the impact of matrix complexities on the quantification of naphthenic acids in OSPW using low-resolution GC-MS. Analytical alternatives available to overcome problems inherent in low-resolution GC-MS analysis, and suitable for application in a world class environmental monitoring program, are also discussed.

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### **Abstract 30**

#### **Toxicity Mapping using Passive Air Sampling for Polycyclic Aromatic Compounds**

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Released from pyrogenic and petrogenic sources, polycyclic aromatic compounds (PACs) are ubiquitous and potentially carcinogenic and mutagenic. Once emitted to the air, some PACs undergo reactions with atmospheric oxidants converting the unsubstituted polycyclic aromatic hydrocarbons (PAHs) into species such as nitrated PAHs (NPAHs) and oxygenated PAHs (OPAHs). These PAH derivatives are of interest as some exhibit higher mutagenicity than their parent PAHs<sup>1</sup>. PAH inhalation exposures have often been associated with lung cancer risk and other health concerns<sup>2</sup>.

The concept and preliminary results of toxicity mapping of air, which links passive air monitoring with toxicity assessment, will be presented. The passive air samplers, deployed in Toronto, Ontario during December 2013 to February 2014 and in the Athabasca oil sands region in Alberta during April to May 2014, were quantitatively analyzed for PACs, NPAHs, and OPAHs. Toxicity of the air extracts was determined by the bacterial mutation assay using Salmonella strain TA98 with and without metabolic activation, and by the cytotoxicity assay using human lung epithelial A549 cells. Spatial distribution of PAHs and PAH derivatives in air in the oil sands correlated with mutagenicity and cytotoxicity, respectively. The mutagenic and cytotoxic responses for Toronto and the oil sands sites were found to be in the same range. In addition, a comparison between two types of polyurethane foam (PUF) disk samplers - conventional passive air sampler (PAS) and passive dry deposition sampler (PAS-DD) - was carried out in this study. The PAS and PAS-DD samplers were co-deployed at the same location in Toronto. The PAS-DD sampler was shown to capture higher amounts of particle-bound PACs (more

potent mutagens). The higher proportion of particle-bound PACs resulted in higher mutagenicity and cytotoxicity of the PAS-DD samples relative to the PAS samples.

The results of this study show promise for applying toxicity mapping as a complement to air concentration mapping that is typically done in spatial air monitoring studies using passive air samplers. Toxicity mapping is a pragmatic approach for assessing risks to humans and wildlife as it considers the entire chemical mixture that includes known and unknown chemicals and considers numerous toxic endpoints, in addition to mutagenicity and cytotoxicity that were evaluated in this study.

<sup>1</sup>Yang, X.-Y.; Igarashi, K.; Tang, N.; Lin, J.-M.; Wang, W.; Kameda, T.; Toriba, A.; Hayakawa, K., Indirect- and direct-acting mutagenicity of diesel, coal and wood burning-derived particulates and contribution of polycyclic aromatic hydrocarbons and nitropolycyclic aromatic hydrocarbons. *Mutat. Res-Gen. Tox. En.* **2010**, *695*, (1-2), 29-34.

<sup>2</sup>Ishikawa, H.; Tian, Y.; Piao, F.; Duan, Z.; Zhang, Y.; Ma, M.; Li, H.; Yamamoto, H.; Matsumoto, Y.; Sakai, S.; Cui, J.; Yamaguchi, T.; Yokoyama, K., Genotoxic damage in female residents exposed to environmental air pollution in Shenyang city, China. *Cancer Letters.* **2006**, *240*, 1, 29-35.

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### Abstract 32

#### **Methylmercury in sediments from shelf and continental slope at Campos Basin near Rio de Janeiro, Brazil**

Beatriz Araujo, Brian Dimock, Marcelo Almeida, Ana Paula Falcão, Carlos Eduardo Rezende  
Trent University, Peterborough, Ontario, Canada

This study aimed to establish the distribution of Total Hg (THg) and methylmercury (MMHg) in surface sediments of three transects (at 25 - 3000m water depth) on the continental shelf and slope in Campos Basin, RJ, Brazil. The silt/clay, organic carbon (OC) and Sulfur (S) contents ranged from 0.05 to 95%, 0.07 to 1.43 % and 0.01 to 0.18% for all transects, respectively. THg and MMHg average concentrations in the transect A were 19.8 (3.3 – 33.2) ng.g<sup>-1</sup> and 0.18 (0.02 – 0.40) ng.g<sup>-1</sup>; in transect D 23.5 (3.3- 51.6) ng.g<sup>-1</sup> and 0.11 (0.03 – 0.29) ng.g<sup>-1</sup>; and in transect I 26.1 (1.6 – 38.2) ng.g<sup>-1</sup> and 0.09 (0.02 – 0.16) ng.g<sup>-1</sup>, respectively. Except for the A transect, MMHg was higher in the upper slope (400-1900m) compared to the shelf region. The A transect is located in an upwelling area. These areas are characterized by high productivity and consequently, availability of organic matter which may contribute to higher methylation rates. The MMHg concentrations found in this study are within the range of values reported for other areas in oceanic regions.

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### Abstract 34

#### **Polycyclic Aromatic Compounds (PACs) in Air across the Athabasca Oil sands region: Assessing Ecosystem Exposure**

Tom Harner  
Air Quality Processes Research Section, Environment Canada, Toronto, Ontario, Canada

In the Athabasca oil sands region (AOSR), polycyclic aromatic compounds (PACs) are released from a variety of petrogenic and pyrogenic sources and are of concern due to their potential to elicit mutagenic and carcinogenic effects. Under the Canada/Alberta Joint Oil Sands Monitoring Plan (JOSM) PACs are monitored to support the overall JOSM objective of assessing the cumulative effects of the oil sands operations on ecosystem and human health. Monitoring for these pollutants has been established using a network of 17 passive sampling sites and 3 active air sampling sites. The key objectives include, i.) to assess spatial and temporal trends of PACs in air to better understand cumulative environmental effects associated with mining activities, ii.) to estimate the atmospheric deposition of PACs, and iii.) to provide data on PACs in air to support related studies on PACs in the AOSR. This presentation will give an overview passive air sampling and the results of the air monitoring program for PACs and how the data are being used to support deposition modelling and avian effects monitoring. Information will also be presented on a toxicity mapping technique which involves applying the passive air sampler extracts to in-vitro assays. Preliminary results show that the spatial distribution of PAH and PAH derivatives concentrations in air correlate with mutagenicity and cytotoxicity, respectively.

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## Abstract 35

### The occurrence of HBCD and its diastereomers in Toronto urban and global air

Cassandra Rauert, Lutz Ahrens, Mahiba Shoeib, Tom Harner

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Hexabromocyclododecane (HBCD) is a globally used brominated flame retardant primarily applied in building thermal insulation, back coatings of fabric and furniture, and in high intensity polystyrene (HIPS) for electronic equipment. In Canada, it is a priority chemical under the Chemicals Management Plan and in 2013 was added to Annex A (decision SC-6/13) of the Stockholm Convention, for reduction of use internationally. Concentrations in indoor air and dust provide an HBCD source for outdoor air contamination, however reported outdoor air concentrations are reduced to that determined indoors. The majority of outdoor air studies have analysed total HBCD content rather than determining the contribution of the three main diastereomers present in the HBCD technical formulation (hence the three primarily found in the environment). New information on HBCD isomers in outdoor air may provide insight to the emission sources, transformation and atmospheric transport of the HBCD diastereomers. The present study has developed a sensitive and selective method for the determination of  $\alpha$ -,  $\beta$ - and  $\gamma$ -HBCD utilising Ultra Performance Liquid Chromatography tandem Mass Spectrometry (UPLC-MS/MS). Active air samples from an urban air study in Toronto were analysed and concentrations ranging 0.01-3.43  $\text{pg}/\text{m}^3$  ( $\Sigma\text{HBCD}$ ) were determined in high volume samples collected bi-weekly over 24-hour sampling periods. The (%) diastereomer contributions to the total HBCD content were shown to be highly variable for the  $\alpha$ - and  $\gamma$ - diastereomers, and is consistent with the few previous outdoor air studies that have analysed diastereomer contributions. Finally, passive samples from the Global Atmospheric Passive Sampling (GAPS) network were analysed with the developed methods to provide an indicator of global outdoor air concentrations. Detection was low with HBCD diastereomers only detected in 20% of the samples and concentrations ranged from 0.06-10.2  $\text{pg}/\text{m}^3$  ( $\Sigma\text{HBCD}$ ).

## Abstract 37

### Arsenic and selenium in the lower Athabasca River, Alberta

Mark Donner<sup>1</sup>, William Shotyk<sup>1</sup>, Kevin Francesconi<sup>2</sup>, Tariq Siddique<sup>1</sup>

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<sup>2</sup> University of Graz, Institute of Chemistry - Analytical Chemistry, Austria

Industrial development of the Athabasca bituminous (oil) sands has raised concern for water quality in the Athabasca River. Along with a variety of other potentially toxic elements, As and Se have been identified as possible contaminants being emitted to the river by industrial activity. To investigate the presence of As and Se in the Athabasca River, surface water samples were collected at 13 locations, varying in distance from oil sands facilities. Additional samples were also collected from groundwater, tributaries and springs to examine a variety of aquatic inputs to the river. Filtered samples (< 0.45 µm) were analyzed for total concentrations using a combination of ICP-MS (iCAP Q) and HG-AFS (Millennium Excalibur). The behaviour, fate, and ecological significance of As and Se, however, depend not only on their concentrations but also on their oxidation states. With this in mind, chemical speciation analyses were performed using ion chromatography (ICS-5000) coupled with ICP-MS.

Concentrations of total dissolved As in the Athabasca River were uniform (0.37 +/- 0.01 µg/L, n=14), with no increasing trend, or change in inorganic species downstream of oil sands activity. The most abundant chemical form was arsenate, As(V), with a small fraction of arsenite, As(III). Arsenic in tributary waters showed greater variability between sites (0.45 to 0.95 µg/L), while groundwater samples contained higher overall values (2 to 20 µg/L), predominantly as As(III). Two sites, one near a tailings pond and the other near an upgrader, showed elevated concentrations of As in shallow groundwater aquifers, but there was no measurable influence on downstream surface water concentrations. Selenium concentrations were low in all water samples (averaging < 0.12 µg/L) with no significant spatial variations in concentrations. Concentrations in groundwater (range 0.07 to 0.36 µg/L) were greater than in surface waters. The ultra-low concentrations of Se highlights the need for specialized methods to accurately determine species at ng/L (ppt) concentrations. Despite the obvious industrial activity, there are no discernible impacts on either As or Se in the Athabasca River. More research however, is required to better understand the transformations taking place at the groundwater-surface water interface.

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## Abstract 38

### Analysis of Pesticides and Dioxins in Food on Modern Triple Quad GC-MS/MS

Alexander Semyonov, Dwain Cardona, Amit Gujar  
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The power of the modern QqQ GC-MS/MS instrumentation will be explored on the analysis of over a thousand of pesticides quantified at single-digit ppb level. Advanced tools for rapid method creation, optimization, and maintenance with newly important pesticides will be demonstrated to save ten to hundred fold analyst time and eliminate human errors. Comparison of the data in the analysis of dioxins

on classical, U.S. EPA required magnetic sector HRAM GC-MS with quantitation of dioxins on QqQ as well as on the newly available Q Exactive HRAM GC-MS/MS based on Orbitrap (TM) technology. Value/performance and instrumentation cost vs. results confidence will be discussed.

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## **Abstract 39**

### **Moss and peat as monitors of past, present, and future rates of atmospheric dust deposition**

Gillian Mullan-Boudreau, William Shotyk

University of Alberta, Edmonton, Alberta, Canada

Open pit mining of the Athabasca bituminous sands generates enormous quantities of mineral dusts. One measure of the success of reclamation in this area will be the reduction of dust emissions to pre-mining, or natural “background” levels. Mining is by no means the only industry with anthropogenic emissions of atmospheric dusts: forest-clearing (agriculture and forestry) and construction (airports, roads, towns and other infrastructure) also contribute to the burden. In the pre-anthropogenic period, dust deposition was primarily affected by climate change with some periodic volcanic inputs, indicating that northern Alberta may have considerable variation in the rates and sources of dust deposition throughout time. The purpose of this research is to reconstruct present and past rates of atmospheric dust deposition, using moss and peat, to identify the natural “background” rates for which future reclamation practices shall strive to achieve.

Present-day rates of dust deposition are being obtained by ashing Sphagnum moss from ombrotrophic bogs surrounding the bituminous sands region. The plants growing in ombrotrophic bogs receive all their nutrients and water from the atmosphere, making any dusts a key component in mineral uptake. The concentrations of acid-insoluble ash (AIA) combined with surrogates of mineral matter concentrations, namely Al, Ba, Sc, Ti and Th, are then analyzed. Past rates of dust deposition are being obtained using the same techniques, except applied to peat cores from the bituminous sands region, which have been age-dated using  $^{210}\text{Pb}$  and  $^{14}\text{C}$ . Electrical conductivity and pH were also measured from the porewater of the peat cores to establish the thickness of the ombrotrophic layers. Finally, the morphologies of the mineral particles are also being studied using scanning electron microscopy imaging and chemical composition analysis.

Results from moss samples show a wide range of dust particle morphology and size that tends toward larger particles, e.g. 8-100  $\mu\text{m}$ , which indicates localized deposition. The electrical conductivity and pH of the peat cores reveal ombrotrophic layers ranging from 18 to over 100 cm. Chemical analyses of the particles using the x-ray fluorescence spectrometer of the SEM will help to identify the predominant dust sources. Particle size analyses will also be performed on the AIA fraction of the moss and peat.

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## **Abstract 40**

### **Thermal mobilization and Arsenic: implications for the oil sands industry**

Jon Fennell

Integrated Sustainability Consultants Ltd., Calgary, Alberta, Canada

The oil sands of western Canada represent a significant global hydrocarbon resource. The deposits themselves are located in three main areas of Alberta - Peace River, Cold Lake, and Athabasca (northern and southern regions). The largest of these is the Athabasca Oil Sands deposit, which covers an area of roughly 93,000 km<sup>2</sup> and constitutes 66% of the total oil sands area. Upwards of 97% of these bitumen-saturated sands are only accessible by in situ extraction techniques, which currently employ the injection of high temperature steam to reduce bitumen viscosity and facilitate recovery to surface. As the steam is conveyed down the dedicated steel-cased wells, and hot fluids are returned to surface, heat is transferred to the surrounding sediments and associated porewater via conduction.

A field and laboratory study was designed to assess the risk of thermal mobilization and revealed that changes to geochemical conditions do occur as a result of subsurface heating. Of particular note was the release of arsenic (among other constituents) into a pristine aquifer at concentrations well in excess of guidelines for the protection of human health and aquatic life. Coincident changes in iron, manganese, pH, redox, dissolved gases, and stable isotopes supported reductive dissolution and surface-controlled reactions as the mechanism. Calculated activation energies for arsenic release ranged from 2 to 12 kcal/mol consistent with physical desorption, mineral dissolution via surface reaction control, and microbially-assisted reactions. Sequential soil extractions of aquifer sediments indicated amorphous Fe and Al hydrous oxides as the controlling fraction, implicating clays and possibly oxide minerals as the source and sink. Mineral characterization and geochemical modeling supported this conclusion, revealing Fe-rich smectite clay as the dominant player. Differential movement of mobilized constituents under advective groundwater flow confirmed attenuation of arsenic with a retardation factor of 1.6. Bench-scale testing supported these findings.

The results from this study have helped place the risk of thermal mobilization into context through enhanced understanding of mobilization mechanisms, as well as transport and fate characteristics. Although this risk may vary by location, based on unique geochemical conditions, natural attenuation mechanisms present themselves as an ameliorating factor to this risk.

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## **Abstract 41**

### **Reagent-Free Ion Chromatography (RFIC) and its Application in Environmental Ion Analysis**

Joachim Weiss

Thermo Fisher Scientific GmbH, Dreieich, Hessen, Germany

In the almost forty years that encompass its birth and development, ion chromatography (IC) has undergone enormous changes. While in its earliest embodiments IC was focused primarily on the analysis of inorganic anions, today IC has an important role in the analysis of organic and inorganic anions and cations. For the majority of applications, suppressed conductivity detection represents the most versatile detection system augmented today. Other detection methods include UV/Vis detection,

various application forms of amperometry, charge detection as well as element-specific (IC-ICP), and mass-selective detection (IC-MS).

One of the major breakthroughs in the development of Ion Chromatography (IC) was the introduction of Reagent-Free IC in 2003, a combination of membrane-based technology and electrolysis to generate high-purity acid or base with only deionized water as the carrier. Because eluent concentration is controlled electrically, isocratic or gradient applications are enormously simplified. In addition, RFIC facilitates sub-ppb level anion and cation analysis by direct injection. Traditionally, eluents in ion chromatography are prepared off-line from reagent-grade chemicals. However, off-line preparation often introduces contaminants that can cause undesirable chromatographic baseline artifacts. When working with hydroxide eluents in anion exchange chromatography, for instance, irreproducible retention times of target analytes are observed due to carbonate contamination. By generating hydroxide eluents on-line via electrolysis, eluent contaminants such as carbonate are eliminated, which improves baseline stability during gradient runs.

The concept of RFIC is supported by a series of hydroxide-selective anion exchangers as well as weak acid cation exchangers for a variety of applications. Compliance monitoring of common inorganic anions and cations in all kinds of aqueous samples is one of the most important applications of RFIC. While suppressed conductivity detection is still the most important detection method for standard inorganic anions, cations, and amines, UV detection is often used for trace analysis of nitrite, nitrate, bromide, and iodide. Toxic anions such as sulfide and cyanide are usually detected electrochemically. However, in recent years there has been considerable interest in the development of IC methods to meet regulatory requirements for analytes other than common inorganic anions, including disinfection by-products such as bromate, chlorite, and haloacetic acids. Many of these methods require the use of large injection volumes, high-capacity columns, and advanced detection schemes such as IC/ICP-MS or post-column derivatizations with UV/Vis detection to meet current regulatory objectives. The combination of IC with mass-selective detection is emerging as an important tool for the analysis of trace ionic species in aqueous samples, as it provides increased specificity and sensitivity in comparison to traditional detection methods.

In this presentation, the principles of RFIC are described and the advantages in isocratic and gradient IC separations of environmentally relevant anions and cations are demonstrated.

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## **Abstract 42**

### **A retrospective look at Nevada Test Site fallout: studies with plutonium isotopes**

Elizabeth Ketterer

Metropolitan State University of Denver, Colorado, USA

The US Government conducted atmospheric nuclear tests at the Nevada Test Site (NTS), located in the desert ~ 100 km to the northwest of Las Vegas. These tests released radionuclides (fission products, actinides, and activation products) into the troposphere; “fallout” was deposited locally and regionally onto the terrain. Previous studies of the US Government and other researchers have identified NTS fallout, principally in the states of Nevada and Utah. Previous studies spurred legislative action; in 1982, the US Congress passed the Radiation Exposure and Compensation Act (RECA), commonly referred to as the “Downwinder” settlement.

Our recent work has sought to validate, retrospectively, the distribution of NTS fallout in various parts of the continental US. An effective tool in these studies is plutonium isotope ratio measurements. The atom ratio  $^{240}\text{Pu}/^{239}\text{Pu}$  is  $\sim 0.03\text{--}0.07$  for NTS fallout; in contrast, “background” Pu from globally deposited stratospheric fallout has a  $^{240}\text{Pu}/^{239}\text{Pu}$  of 0.18. Plutonium atom ratios are measured by mass spectrometry; the most practical technique to use is inductively coupled plasma mass spectrometry (ICPMS). Typically, samples are dissolved or leached; Pu is separated by ion exchange or extraction chromatography, and concentrated Pu solutions are analyzed using sector or quadrupole ICPMS using a high-efficiency sample introduction system.

We have identified NTS Pu in various locations in the continental US. This presentation will focus, in particular, on the studies of Pu in “upwind” locations in the eastern California Sierras. A dominant Nevada Test Site Pu contribution has been found in soils of Inyo and Mono Counties in California, locations situated to the west and northwest of the NTS, opposite of the prevailing wind direction. The top 10 cm horizons of soils from 17 montane locations exhibit  $^{240}\text{Pu}/^{239}\text{Pu}$  ratios systematically lower than the 0.180 value of stratospheric fallout (range 0.054 - 0.158, average = 0.089, standard deviation = 0.021, n = 99). Nevada tests account for 11 to 86% of the total  $^{239+240}\text{Pu}$  activity in individual samples. The isotope ratios  $^{241}\text{Pu}/^{239}\text{Pu}$  and  $^{242}\text{Pu}/^{239}\text{Pu}$  co-vary with  $^{240}\text{Pu}/^{239}\text{Pu}$ , demonstrating a distinct pattern of two-component mixing between Nevada and stratospheric fallout. Values for the minor isotope ratios  $^{241}\text{Pu}/^{239}\text{Pu}$  and  $^{242}\text{Pu}/^{239}\text{Pu}$  in California soils were found to lie along the same mixing lines described in previous studies of Nevada and Utah soils. This work illustrates the widespread presence of Nevada Test Site Pu in an “upwind” direction, despite the US Government’s policy of having conducted tests at times when winds would have carried fallout away from California. Although residents of Inyo and Mono Counties are not currently eligible for compensation under the RECA, this study reveals an upwind Pu depositional pattern northwest of the NTS that has not been previously recognized.

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## Abstract 43

### Fate of silver nanoparticles in lake water

Holger Hintelmann

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This study aimed to evaluate the persistence and transformation of silver nanoparticles (AgNP) in natural lake water using various analytical techniques. Studies were conducted by spiking mesocosms deployed at the Experimental Lakes Area (ELA), ON, Canada with AgNPs at a nominal concentration of  $45\ \mu\text{g L}^{-1}$ . Samples were collected for analysis by centrifugal ultrafiltration, CPE, spICP-MS, and AF4-ICP-MS to monitor changes in AgNP agglomeration and dissolution over time. Total Ag levels in the two mesocosms declined relatively slowly, with a  $t_{1/2}$  of  $\sim 20$  days. There was no evidence of extensive agglomeration of AgNPs. The stability of AgNPs may have been due to the low ionic strength and moderate concentrations of dissolved organic carbon (DOC) in the lake water. CPE, spICP-MS, and AF4-ICP-MS analysis all indicated that there was dissolution of AgNPs over time. The results from this study emphasize that multiple analytical techniques are required in order to fully characterize AgNP transformations in the aquatic environment and to overcome the analytical limitations of each method.

## Abstract 44

### The Application of ICPMS in Nuclear Industry - Determination of Pu Isotopes in Urine Sample

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Pu isotopes are the major contributors for alpha dose for nuclear industry workers. The presentation describes the applications of sector field Inductively-coupled plasma mass spectrometry (SF-ICPMS) in nuclear industry for Pu isotopes (<sup>239</sup>Pu, <sup>240</sup>Pu, <sup>242</sup>Pu) analysis rather than conventional method of alpha spectrometry. SF-ICPMS has gained the use in dosimetry due to its greater sensitivity, rapid throughput capability and flexibility in handling for emergency response in nuclear industry. It is a fast screening method for workers in nuclear stations.

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## Abstract 45

### Characterizing the size and optical properties of Athabasca R. dissolved organic matter: towards bitumen-based optical tracers and measurement of metal partitioning

Chad W. Cuss, William Shotyk, Ehsanul Hoque, Beatriz Bicalho  
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Given extensive natural bitumen deposits and several other sources of distinct organic matter (e.g. peat bogs, saline springs), along with industrial, commercial, and residential developments along its banks, the Athabasca River constitutes a unique and important aquatic system. Dissolved organic matter (DOM) governs the mobility and toxicity of potentially harmful pollutants such as naphthenic acids and “heavy” metals, and its reactivity is determined by structure and composition. Thus, the non-destructive characterization of organic matter and associated pollutants in the Athabasca R., and the source waters that feed it, are of significant importance. To adequately describe the distribution and speciation of DOM and associated pollutants, such characterization should be non-destructive to both structure and composition, requiring gentle experimental treatments and analysis under ambient conditions of pH and ionic strength. Asymmetrical flow field-flow fractionation (AF4) gently separates nanometer-scale particles along a size continuum based on their diffusion properties, and allows analysis under a wide range of pH values and ionic strengths.

This presentation will focus on the application of AF4 with on-line UV-visible and fluorescence detectors (AF4-UV-FLD) for the characterization of DOM from the Athabasca R. and its source waters, conducted in the ultra-clean, metal-free SWAMP laboratory at the University of Alberta. The particulars of the method will be discussed, and data from the characterization of both natural bitumen deposits and the Athabasca R. will be presented. Particular attention will be paid to the potential for isolating bitumen-based optical tracers by applying the advanced chemometric method parallel factor analysis (PARAFAC) to the fluorescence excitation-emission matrices of size-separated material. Early results from the analysis of metal-DOM distributions measured using the on-line coupling of AF4-UV-FLD to sector-field ICP MS will also be discussed.

## Abstract 47

### Concentrations of heavy metals (Ag, As, Cd, Pb, Sb, Tl) in Athabasca bituminous sands are comparable to crustal values

Beatriz Bicalho, Iain Grant-Weaver, Gillian Mullan-Boudreau, Muhammad Babar Javed, William Shotyk  
University of Alberta, Edmonton, Alberta, Canada

Development of the oil sands industry in northern Alberta has raised legitimate concerns about pollution and disruption of existing ecosystems. Contaminants may be released to the environment during mining, transporting and upgrading of bituminous sands, or through the generation of by-products, either solid (coke) or liquid (process water). In respect to potentially toxic heavy metals such as Ag, Cd, Pb, Sb and Tl, much attention has been placed on the study of receptors, but very little attention has been paid to the sources. While natural, geochemical enrichments of V, Ni and Mo in bitumen have been well known for decades, much less is known about the abundance of heavy metals. As a first step to better understand the abundance and occurrence of these elements, samples of bituminous sand were collected from the banks of the McKay River and the Athabasca River (where it receives the Pierre River).

Organic and mineral phases were separated using mass spectrometry grade organic solvents and labware made either of high purity quartz or Teflon<sup>®</sup> were used for preparation and handling. Trace metal concentrations were determined in the ultraclean SWAMP lab using ICP-MS (ICAP Qc) following high pressure/temperature acid digestion. About 15% (w/w) of the bituminous sands was removed as organic matter along with 50% of total V, Ni and Mo. SEM analysis confirmed C depletion and silicate enrichment in the bituminous sands after the organic extraction. Although the extracts were filtered on quartz wool, substantial amounts (50%) of Th and Y were found in the organic extracts. Because Th is hosted in mineral phases such as monazite and zircon which are very resistant to chemical attack, their abundance in the organic fraction suggests that some mineral particles were not retained in the filtration system. Heavy metals such as Ag, Bi, Cd, Cu, Pb and Tl were found mainly (75 - 85%) in the mineral fraction. Taken together, some part of the heavy metal inventory of the organic fraction may also be in a mineral form. The total concentration of heavy metals in the raw (untreated) bituminous sands varied, but were on the order of 6 µg/g Pb, 5 µg/g Cu, 2 µg/g As, and 150 ng/g Tl; Ag, Bi, and Cd were not greater than 50 ng/g. All of these concentrations, however, are comparable to the Upper Continental Crust (Wedephol, 1995) and soil (Bowen, 1979). In other words, none of the heavy metals are enriched in the bituminous sands themselves.

The focus of the work presented here was the development of appropriate analytical methods for measuring heavy metals in the organic fraction of bituminous sands, while avoiding contamination, and the study was limited to two samples. Clearly, this work needs now to be extended not only to more samples from the Athabasca Bituminous Sands, but also from the Cold Lake and Peace River deposits as well. Despite this, the results obtained thus far offer a plausible explanation for the lack of enrichment of heavy metals in *Sphagnum* moss from peat bogs surrounding the open pit mines and upgraders: there is no significant enrichment of heavy metals in the bituminous sands themselves.

## Abstract 48

### **An arsenic speciation method optimized for arsenite, arsenate and DMAA species in river water and sediment using IC-ICP-MS**

Muhammad Babar Javed, Tariq Siddique  
University of Alberta, Edmonton, Alberta, Canada

A comprehensive arsenic (As) speciation protocol was optimized for arsenite ( $\text{As}^{\text{III}}$ ), arsenate ( $\text{As}^{\text{V}}$ ) and DMAA species in river water and sediment samples. For chemical quantification of the species, analytical method was optimized using ion chromatography technique coupled with quadrupole inductively coupled plasma mass spectrometer (IC-ICP-MS). An anion exchange column (Dionex AS-7: 4 mm I.D., 250 mm length), 2.5 mM to 50 mM nitric acid ( $\text{HNO}_3$ ) mobile phase and a gradient elution were used in IC to resolve the As species, which were individually quantified using ICP-MS. The developed speciation method was equally sensitive ( $\sim 1200$  cps ppb $^{-1}$ ) for all three As species with 20 to 50 ng L $^{-1}$  (ppt) detection limits.

The effect of pH (2 to 10; pH 2 adjusted with both  $\text{HNO}_3$  or HCl to determine the effect of acids, pH 4 to 10 adjusted with NaOH) on stability of  $\text{As}^{\text{III}}$ ,  $\text{As}^{\text{V}}$  and DMAA species in deionized water under both aerobic and anaerobic conditions over time (12 and 24 h) was also studied. The results showed that lowering the pH to 2 with HCl only preserved all three As species in deionized water under both aerobic and anaerobic conditions up to 24 h.

To preserve the  $\text{As}^{\text{III}}$ ,  $\text{As}^{\text{V}}$  and DMAA species in river water, water samples were collected from the North Saskatchewan river and common preservation methods such as acidification (pH 2 using HCl), filtration (0.2  $\mu\text{m}$  and 0.45  $\mu\text{m}$ ), and complexation (using EDTA pH 3.2 adjusted with acetic acid) were applied in the field. The results showed that acidification using HCl, 0.2  $\mu\text{m}$  filtration or EDTA complexation were effective methods to preserve the distribution of all three As species in river water containing  $\sim 1$   $\mu\text{g L}^{-1}$  total As for 7 days. In addition, to determine the As species in sediment an extraction method was also optimized where 0.1 M  $\text{NaH}_2\text{PO}_4$  extraction solution at different pH (pH 2, 5, and 7) and different extraction time (6 to 24 h) were tested. The results showed that 0.1 M  $\text{NaH}_2\text{PO}_4$  (pH 5) extraction of sediment for 18 h was optimum extraction conditions to extract the As species without significant transformation. The study provides a complete protocol for speciation of  $\text{As}^{\text{III}}$ ,  $\text{As}^{\text{V}}$  and DMAA in river water and sediments.

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## Abstract 49

### **Maintaining the Power Grid - a Simplified and Efficient Approach to Routine Monitoring of Transformers Using Automated Solid Phase Extraction Furan Testing in Transformer Oil**

Vinson Leung, Michael Ebitson, Jim Fenster, Zoe Grosser, Toni Hofhine, Jean-Luc Brousseau  
Horizon Technology, Salem, New Hampshire, USA

The average age of many large power transformers around the globe is reaching 40 or more years. Due to cost and today's growing demand for power, many countries are choosing to maintain rather than replace transformers. Maintaining power transformers involves routine testing of the transformer oil to monitor the furan levels present, with the intent to provide assurance of both transformer safety and

power supply reliability to communities across the globe. Unexpected failures of power transformers can lead to significant costs - a replacement transformer, environmental cleanup from the failure, as well as potential financial penalties for not delivering power to communities. With these financial and safety factors in mind, testing of aging power transformers is increasing significantly using the traditional manual ASTM D5837 solid phase extraction procedure of the sampled transformer oil. Frequency and number of samples tested is growing, leaving many power companies and contract laboratories searching for new automated solutions that use the ASTM procedure to offer simplicity and efficiency versus hiring and training more laboratory chemists. This application presents data to compare automated and manual solid phase extraction ASTM D5837 testing procedures to offer a simplified and more efficient approach for workflow, accuracy, precision, and operator time.

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## **Abstract 50**

### **Rugged Method 625 for Wastewater Extraction by Solid Phase Materials**

Jean-Luc Brousseau, Zoe Grosser, William Jones, David Gallagher, Michael Ebitson  
Horizon Technology, Salem, New Hampshire, USA

US EPA method 625 is a general semivolatile method for wastewater analysis applied to a large suite of target analytes. Although method 625 was developed a number of years ago, through the EPA Office of Water, Office of Science and Technology, the method has been updated several times. As new technology is developed either for the determinative measurement or, earlier in the analysis process, for the sample preparation, data must be collected to demonstrate that the new technology is compliant and reproducible.

Two small round-robin studies using multiple vendor products and solid phase extraction (SPE) materials in a variety of laboratories have been run to demonstrate the compliance of SPE with method requirements. The first study relied on the quality control in the existing method to catch when the equipment or sorbent did not work properly. In the second study, the choice of surrogates was enlarged to ensure that errors not caught with the criteria in the older version of 625 would be identified in this version.

This paper will discuss the performance of SPE in general for method 625 and the specific performance of a disk used with a single pass of acidified water through it rather than a pass with basified water and a second pass with the same water, now acidified, which is typical for liquid-liquid extraction. Recoveries of a large suite of compounds from a variety of matrices and laboratories will be examined and the effect of surrogates will be considered. The results from the first round robin demonstrated recoveries from 70-130% of most all the acid/base/neutral/pesticides chosen for the study using a multi-mode disk adsorbent. These results will be compared to results from the second study and comparisons of large and small volumes of sample, different surrogate mixes and interlaboratory performance will be discussed.

## Abstract 51

### Characterizing organic compositions in snow and surface water in the Athabasca oil sands region

Yi Yi<sup>1</sup>, Jean Birks<sup>1</sup>, Sunny Cho<sup>2</sup>, John J Gibson<sup>1</sup>

<sup>1</sup> Alberta Innovates – Technology Futures, Victoria, British Columbia, Canada

<sup>2</sup> Alberta Environment and Sustainable Resource Development, Edmonton, Alberta, Canada

Fourier Transform Ion Cyclotron Resonance Mass Spectrometry (FTICR MS) was applied to understand the distribution and composition of polar organics in the Athabasca oil sands region (AOSR). Snow, streams (Athabasca River and its tributaries) and lake samples were collected in 2012 and 2013. The overall goal of the research was to understand potential impact of atmospherically-derived substances on surface water, with a focus on application of organic compositions for better understanding of source and transport of organics. The profiling results from two consecutive years consistently demonstrate that snow samples are compositionally different from surface waters in the Athabasca Oil sand region. It is also interesting to note that several subgroups of snow samples can be readily identified based upon organic compositions, which are possibly affected by, but not limited to, the distance from the sampling sites to the major emission centres of the oil sands industry. Along the main stem of the Athabasca River, organic compositions appear to show systematic changes from upstream (Athabasca River at Athabasca) to downstream of industrial development (Athabasca River at the confluence with the Firebag river). The upstream Athabasca River sites show compositional similarities to snow, while the downstream Athabasca River sites are more similar to local lakes and tributaries. This compositional contrast likely indicates the relative role of regional snowmelt contributions to the Athabasca River in early spring vs local inputs generated via catchment runoff processes in the summer.

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## Abstract 52

### Trace and ultra-trace analysis of chromium (VI) by ion chromatography tandem mass spectrometry

Vasile Furdui<sup>1</sup>, Stefanie Maedler<sup>1</sup>, Teresa Switzer<sup>1</sup>, Fengrong Sun<sup>1</sup>, Cindy Tat<sup>1</sup>, Robert Tooley<sup>1</sup>, Matt Pamuku<sup>2</sup>, H. M. Skip Kingston<sup>3</sup>

<sup>1</sup> Ontario Ministry of the Environment and Climate Change, Toronto, Ontario, Canada

<sup>2</sup> Applied Isotope Technologies (AIT) Inc., Pittsburgh, Pennsylvania, USA

<sup>3</sup> Duquesne University, Pittsburgh, Pennsylvania, USA

Hexavalent chromium [Cr(VI)] has been routinely quantified by colorimetry or ion chromatography (IC) separation with post column derivatization using 1,5-diphenylcarbazide. While these approaches work well for colorless samples with Cr(VI) content higher than 1 ug/L, significant interferences can occur in samples with high content of dissolved organic carbon (DOC) or in the presence of similar anions like molybdate and tungstate. New methods for analyzing trace level Cr(VI) were developed using ion chromatography-tandem mass spectrometry (IC-MS/MS), offering a complementary approach to the existing analytical techniques. Reaching a detection limit below 5 ng/L allowed us to test the method for Cr(VI) in drinking water samples. The method was also used for background measurements related to the Ring of Fire area, the biggest known deposit of chromite in North America located in Northern Ontario. Samples were recently analyzed, including ground water, surface water, vegetation and

sediment extracts. Influence of various preserving conditions ( $\text{NH}_4\text{OH}$ ,  $\text{NH}_4\text{OH}/(\text{NH}_4)_2\text{SO}_4$  or unpreserved samples), type of quantitation (internal calibration, external calibration and standard additions) and also comparison with results obtained using other Cr(VI) methods will be presented.

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## Abstract 53

### Heavy metals in the Athabasca River: do the bituminous sands have a silver lining?

William Shotyk

University of Alberta, Edmonton, Alberta, Canada

Potentially toxic heavy metals (the chalcophile elements Ag, Cd, Cu, Pb, Sb, Tl and Zn) were determined in water samples ( $< 0.45 \mu\text{m}$ ) from the Athabasca River (AR) along with trace metals known to be enriched in bitumen (V, Ni, Mo, Re) and lithophile elements which are assumed to represent the abundance of inorganic colloids (Al, Co, Cr, Sc, Th, and Y). Water samples were collected at 13 locations between Fort McMurray and the Firebag River, a distance of ca. 125 km, as well as from 5 tributary streams. Water samples were collected using the protocols developed at the University of Heidelberg, Germany for Arctic ice cores and measured using both ICP Q MS (ICAP Qc) and ICP SMS (Element 2 XR) in the new ultraclean SWAMP lab at the University of Alberta. To help put the measured concentrations into perspective, the data are compared to values for surface waters sampled at remote locations in Algonquin Provincial Park, ONT. A second reference level is provided by the values for trace metals from the Elmvale Groundwater Observatory, a purpose-built groundwater sampling station near Elmvale ONT in which artesian groundwaters are sampled within a laminar flow clean air cabinet from surgical stainless steel (316) well.

Concentrations of all potentially toxic trace elements in the Athabasca River are very low, and in general no different from the streams and lakes from ONT. For example, Ag was below the limit of quantification (1.4 ng/l) in all samples using the ICAP Qc, so could only be measured using the Element 2 XR. Cadmium, Co, Cu, Sb and Zn values in the AR are comparable to those from Algonquin Park, but Tl concentrations in the AR are significantly lower. Concentrations of Pb in the Athabasca River are roughly a factor of ten below the levels obtained from the Nippissing River in Algonquin Park. In fact, Pb concentrations in some of the tributary streams of the Athabasca River are no different than ice dating from ca. 5,000 to 8,000 yrs BP from Arctic ice core collected on Devon Island (ie 5 ng/l). To help put this data into perspective, concentrations of Pb and Sb in the AR are dwarfed by the published values for these metals in bottled water packaged in glass and PET plastic, respectively. A large part of the Al, Co, Cr, Sc, Th, and Y in the tributaries is assumed to reflect inputs of organic and inorganic colloids and can explain much of the variation in trace metal concentrations. Taking Pb to reflect inputs of potentially toxic heavy metals generally, the average Pb/Sc ratio of these waters ( $3.1 \pm 1.0$ ,  $n=14$ ) is remarkably similar to the value for the Upper Continental Crust (2.4 according to Wedepohl, 1995), and one-half the value for pre-anthropogenic aerosols from the mid-Holocene as revealed by Swiss peat profiles and Arctic ice cores. Analyses of major ions show pronounced compositional difference between the tributaries and the main stem of the river, and these are attributed to variations in predominant rock types. Given the extremely low concentrations and their independence of distance from industrial activities, anthropogenic inputs of "heavy metals" to the aqueous phase appear to be insignificant. In the main stem of the river, V, Ni and Mo show no pronounced trend in spatial variation, but Re is more abundant downstream (ca. 7 to 14 ng/l) than upstream (ca. 2 to 4 ng/l).

## Abstract 54

### Peat cores from ombrotrophic (rain-fed) bogs in northern Alberta reveal decreasing atmospheric contamination by Pb and other heavy metals

William Shotyk

University of Alberta, Edmonton, Alberta, Canada

In a recent study, *Sphagnum* moss was collected from twenty-one ombrotrophic (rain-fed) peat bogs in the vicinity of and surrounding open pit mines and upgrading facilities of Athabasca Bituminous Sands in Alberta (AB). Mosses are bryophytes, lack roots and receive inputs exclusively from the atmosphere. Compared to the cleanest, ancient peat samples ever tested from the northern hemisphere and dating from the mid-Holocene (ca. 6,000 to 9,000 year old peat from a Swiss bog), none of the potentially toxic heavy metals studied (Ag, Cd, Pb, Sb, Tl) was found to be enriched in the mosses, relative to natural background values.

To provide some perspective about rates and sources of atmospheric deposition in the past, peat cores were collected from the following seven bogs, with the distances from the mid-point between the two upgraders given in brackets: JPH-4 (12 km); Mildred (14 km); McKay (24 km); McMurray (51 km); Anzac (69 km); Utikuma (250 km W SW). One frozen peat core from each of the bogs was sectioned into 1 cm slices. The pH values for the porewaters show that all seven bogs contain an ombrotrophic zone (pH values of 4 being diagnostic). In some profiles (Anzac, Seba Beach, Utikuma) it is clear that the entire peat core (ca. 100 cm long) is within the ombrotrophic zone. At Mildred, in contrast, the ombrotrophic zone is no greater than ca. 20 cm; below this depth there is an abrupt transition to minerotrophic conditions. Plant macrofossils have been identified in every second sample from all seven peat cores to provide a clear indication of the botanical transition from minerotrophic to ombrotrophic peat, but it has also yielded selected plant macrofossils for  $^{14}\text{C}$  age dating. The uppermost sections of the peat cores are also being dated with  $^{210}\text{Pb}$  using ultralow background gamma spectrometry (in progress). The degree of humification is determined using elemental analysis (CHNS-O) and stable isotopes of C and N, and the ash contents measured by combustion. Trace metal concentrations are determined in the ultraclean SWAMP lab using ICP-QMS (ICAP Qc) and ICP-SMS (Element 2 XR), following acid digestion.

At each site, Pb concentrations reveal their greatest values 10 to 40 cm below the surface of the bogs, clearly showing that the zenith of atmospheric Pb contamination occurred some time ago. Similar declines are seen in the concentrations of Ag, Cd, Sb, and Tl. Until the age dating of these cores is completed, a detailed reconstruction of the chronology of deposition is not possible. However, in the absence of  $^{210}\text{Pb}$  dates and considering only the  $^{14}\text{C}$  dates, it appears that atmospheric heavy metal contamination in northern Alberta has been in decline for several decades. Lead concentrations within the top 20 cm of the peat core from Utikuma (our regional "background" site) are on the order of 0.3 mg/kg; this value is not significantly different from the natural background Pb concentrations found in peat from Etang de la Gruère (Switzerland) and dating from ca. 6,000 to 9,000 years BP. The Utikuma core appears to have provided the first evidence of contemporary peat lacking detectable anthrop.

## **Abstract 55**

### **Uncertainty, Error, and Bias in Environmental Measurements and Risk Assessment**

Murray McBride

Cornell University, New York, USA

There is growing concern that recent published research findings have a high level of irreproducibility due to error, bias, misconduct, and in isolated cases, fraud. This disturbing trend is most thoroughly documented in the biological and biomedical sciences. However, environmental sciences may not be immune to this trend, as current scientific practices of data analysis combined with inherent unpredictability of chemical, physical and biological processes in natural and (especially) anthropogenically disturbed environments create a situation likely to produce misleading or false conclusions. I will outline some factors contributing to this problem in environmental science, including heterogeneity of contaminant distribution in soils, and non-normal distribution and heteroskedasticity of data. Using case examples from toxic metal-contaminated soils and crops, I will illustrate pitfalls inherent to the most commonly used standard statistical methods of testing hypotheses.

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## **Abstract 56**

### **Investigating Known Unknowns in the Supercomplex World of Oil Sands Process Water**

Jonathan Martin

University of Alberta, Edmonton, Alberta, Canada

The first attempt to remediate large volumes of oil sands process-affected water (OSPW) is now underway in the world's first oil sands end-pit-lake, called Base Mine Lake (BML). Although naphthenic acids (NAs) are often cited as the major toxic chemical species in the supercomplex mixture of OSPW organics, there is little experimental data to support this. This talk will serve as an overview of an interdisciplinary study using fractionation and effects-directed analysis to identify the major toxic chemical species in OSPW.

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## **POSTER PRESENTATION ABSTRACTS**

### **Abstract 15**

#### **Optimization of dispersive liquid–liquid microextraction coupled with gas chromatography-negative ion chemical ionization mass spectrometry for the determination of pyrethroids in agricultural products**

Yu Chieh Wang<sup>1</sup>, Youn Yuen Shu<sup>1</sup>, JiaLin Wang<sup>2</sup>, Jen-Fon Jen<sup>3</sup>

<sup>1</sup> National Kaohsiung Normal University, Kaohsiung, Taiwan

<sup>2</sup> National Central University, Taoyuan City, Taiwan

<sup>3</sup> Department of Chemistry, National Chung Hsing University, Taichung, Taiwan

In this study Taguchi and response surface methodologies were used for the optimization of dispersive liquid–liquid microextraction (DLLME). Aqueous samples, obtained from homogenization and filtration of agricultural products, were extracted with the optimal conditions: chloroform 70 $\mu$ L (extraction solvent), acetone 300 $\mu$ L (dispersant), and 5% sodium chloride (salt addition concentration) at pH 4 environment with ultrasonic-assisted extraction for 3 minutes. The sedimented phase from DLLME processes was reconstituted to 10  $\mu$ L in acetone solution with hexachlorobenzene as internal standard. 1.0  $\mu$ L of the sample was then injected into gas chromatography-mass spectrometry for the analysis of six pyrethroid pesticides (fenprothrin, fenvalerate, flucythrinate,  $\lambda$ -cyhalothrin, cypermethrin, and deltamethrin) under negative ion chemical ionization (NICI) mode. The method detection was linear at 20-50000 pg/mL with correlation coefficient between 0.991 and 0.996. The method detection limit obtained were between 5-35 pg/mL and repeatability (for both relative standard deviations of the inter- and intra-day analyses) ranged from 3.2 to 8.5%. This method was successfully applied in the determination of 13 real samples, such as beverages, fruits, vegetables, and herbs. The recoveries of standard addition on the real samples ranged between 74% and 130%.

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### **Abstract 20**

#### **Effect of Dwell Time on SP-ICP-MS Data Acquisition Quality**

Aaron Hineman, Chady Stephan  
PerkinElmer Canada, Woodbridge, Ontario, Canada

The characterization, sizing, and quantification of metal-based nanoparticles (NP) in a variety of matrices using single particle-inductively coupled plasma-mass spectrometry (SP-ICP-MS) is becoming increasingly popular due to the sensitive nature of the technique. Nanoparticle events in the plasma are less than 0.5 ms in duration[1]; however current quadrupole-based ICP-MS instruments are limited to instrument dwell times in the millisecond range and have data acquisition overhead that adversely affects data quality. Novel instrument settings and data processing techniques can be used to explore the benefits of continuous data acquisition rates as fast as  $10^5$  Hz (or 10  $\mu$ s dwell times)[2]. This talk presents data on the different effects data acquisition rate has on the quality of data that can be obtained by SP-ICP-MS. The effect of varying the dwell time and its influence on particle integration,

particle counting, particle sizing, and background signal is discussed. Data on identifying the significant instrument settings and their implications on nanoparticle characterization is also presented.

[1] Olesik, J. W., & Gray, P. J. (2012). *J. Anal. At. Spectrom.*, 1143–1155.

[2] Engelhard, C. (2011). *Bioanal. Chem.*, 399, 213-219.

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### Abstract 23

#### Effect of Storage Temperature and Holding Time on the Stability of Inorganic Anions in Drinking Water Samples

Elham Zeinijahromi, Sylvia Tiu, Lorinda Butlin, David W. Kinniburgh  
Alberta Centre for Toxicology, Calgary, Alberta, Canada

Samples from five sources were spiked with  $F^-/NO_2^-$  (0.5 mg/L),  $Cl^-/NO_3^-/SO_4^{2-}$  (5 mg/L), and stored at room temperature or 4°C till the time of analysis. The concentrations of anions were determined using ion chromatography, multiple times, 1-44 days after collection. The aim was to find out a maximum time between sampling and analysis in which the concentration changes were negligible (within  $\pm 10\%$ ). The results showed that  $F^-$ ,  $Cl^-$ ,  $NO_3^-$  and  $SO_4^{2-}$  were stable for 44 days at room temperature and 4°C.  $NO_2^-$  was stable for 2 days at room temperature, while cooling to 4°C extended the holding time to 8 days.

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### Abstract 25

#### Purge-Assisted and Temperature Controlled Headspace Solid-Phase Microextraction for the Determination of Phthalate Esters in Water Samples

Youn Yuen Shu<sup>1</sup>, Yu Chieh Wang<sup>1</sup>, Yun Yun Sun<sup>2</sup>, Robert C Lao, Jen-Fon Jen<sup>3</sup>

<sup>1</sup> National Kaohsiung Normal University, Kaohsiung, Taiwan

<sup>2</sup> Tung Fan Design Institute

<sup>3</sup> Department of Chemistry, National Chung Hsing University

A purge-assisted with temperature-controlled headspace solid-phase microextraction (PATC-HS-SPME) couple to gas chromatography-mass spectrometry (GC-MS) method was developed for the analysis of six phthalates, including benzyl butyl phthalate (BBP), dibutyl phthalate (DBP), bis(2-ethylhexyl)phthalate (DEHP), di-n-octyl phthalate (DnOP), diisononyl phthalate (DINP), and diisodecyl phthalate (DIDP), in aqueous samples. The method is based on the purging of oil bath heated aqueous sample to carry analytes to the headspace that the temperature is controlled by a cooling circulate system. The purpose of this work was to develop a simple, efficient, economical and environmental friendly method for the simultaneous extraction/enrichment of phthalates in water samples. Factors which might affect extraction efficiency, such as type of SPME fiber, extraction time, extraction temperature, headspace temperature, sample agitation rate, salt concentration, and nitrogen purging rate were investigated. Orthogonal experimental design was used for optimizing the parameters that affect the efficiency of PATC-HS-SPME.  $L_9(3)^4$  was chosen according to the three parameters and three levels discussed. Under the optimum condition (DVB/CAR/PDMS 2 cm fiber, extraction temperature at 180 °C, stirring rate of

500 rpm, headspace cooling temperature 20 °C, nitrogen flow rate of 50 mL/min, salt addition 3 g, aqueous pH at 4, extracting time 60 min.). , good linearity was obtained for the analytes with correlation coefficient  $r^2 > 0.992$ . The resulting detection limits ranged from 0.04 to 0.18 ng / mL and the relative standard deviations ranged from 1.4% to 22.4%. The method was successfully applied for the determination of phthalates in aqueous samples.

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### Abstract 31

#### Stability of Polycyclic Aromatic Compounds in Polyurethane Foam-type Passive Air Samplers upon O<sub>3</sub> Exposure

Narumol Jariyasopit<sup>1</sup>, Yongchun Liu<sup>2</sup>, John Liggio<sup>1</sup>, Tom Harner<sup>1</sup>

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<sup>2</sup> State Key Joint Laboratory of Environment Simulation and Pollution Control, Research Center for Eco-Environmental Sciences, Chinese Academy of Sciences, Beijing, China.

Passive air sampling has been widely used in air monitoring and surveillance of persistent organic pollutants (POPs) and other priority chemicals such as polycyclic aromatic compounds (PACs), as it is convenient, inexpensive, and does not require electricity to operate. The sampling media include semipermeable membrane devices (SPMDs), polyurethane foam (PUF) disks, and XAD resin. Because they are easy to handle polyurethane foam (PUFs) disks are commonly used in a number of air monitoring programs. A concern regarding the passive sampling method is the potential reactions of PACs with atmospheric oxidants such as OH radicals, NO<sub>3</sub> radicals, N<sub>2</sub>O<sub>5</sub>, nitrogen oxides, O<sub>3</sub>, etc. during the field deployment. Long deployment time may allow chemicals collected in the sampling media to undergo transformation reactions with the oxidants.

This study investigated stability of PACs in polyurethane foam (PUF) disks upon O<sub>3</sub> exposure in a flow tube chamber at 0% and 50% relative humidity. A wide range of PACs was evaluated by spiking PUF disks with PACs and exposing to O<sub>3</sub> at concentrations that were equivalent to two months exposure, a typical deployment period for these passive air samplers. The results showed that the majority of the PACs were stable at 50% RH upon O<sub>3</sub> exposure. In general, the reactivity of most of the PACs decreased at higher RH, except for the reactive PAHs (acenaphthylene, 2,3-dimethylantracene, 9,10-dimethylantracene, dibenzothiophene, and 2-methyl dibenzothiophene) which demonstrated lower RH dependence. Under conditions of ambient sampling the PUF-based passive sampler is deemed adequate for accurately measuring concentrations in air for most PACs even over extended deployment periods.

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### Abstract 33

#### Seasonal Variability of Total Mercury and Methylmercury in the Amazon floodplain lakes –Brazil

Beatriz Araujo<sup>1</sup>, Brian Dimock<sup>1</sup>, Rodrigo Lima Sobrinho<sup>2</sup>, Marcelo Bernardes<sup>1</sup>, Marcelo Almeida<sup>1</sup>, Carlos Eduardo Rezende<sup>1</sup>

<sup>1</sup>Trent University, Peterborough, Ontario, Canada

<sup>2</sup>Universidade Federal fluminense, Rio de Janeiro, Brazil

The aim of this study is to quantify total mercury (THg) and methylmercury (MMHg) in sediments of five lakes in the central amazon basin during two hydrological seasons of rising waters (RW) and falling waters (FW). The TOC values ranged from 1.3% to 3.5%. THg and MMHg concentrations obtained during the FW season ranged from 978 to 3290 ng g<sup>-1</sup> and 8.39 to 44.2 ng.g<sup>-1</sup>, respectively. During the RW season, THg ranged from 62 to 84 ng. g<sup>-1</sup> and MMHg from 0.62 to 4.78 ng.g<sup>-1</sup>. The extreme hydrological events explain the difference between Hg concentrations in the two seasons. The higher Hg concentration in the FW season can be attributed the huge loads of fine particles washed from the soils and alluvial sediments and transported downstream to the floodplain. On the other hand, the dilution effect promoted in the rising water season of the floodplain areas indicates the remobilization of Hg and, hence, its exportation to other areas.

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#### **Abstract 46**

#### **A Novel Multiwall-Carbon Nanotubes Immobilized Hollow-Fiber Membrane Based Liquid Phase Microextraction for the Analysis of Paclobutrazol in Aqueous Samples by Liquid Chromatography**

Jen-Fon Jen, Vinoth Kumar Ponnusamy

Department of Chemistry, National Chung Hsing University

A novel multiwall carbon nanotubes (MWCNTs) immobilized hollow fiber membrane based liquid-phase microextraction technique was developed for the determination of paclobutrazol in aqueous samples by high performance liquid chromatography with ultra-violet detection. In this study, MWCNTs were immobilized on the inner and outer walls of the polypropylene hollow-fiber membrane segments. Immobilization was accomplished by immersing the hollow-fiber segments into the dispersion of MWCNTs in organic solvent under ultrasonication. The performance and feasibility of the MWCNTs immobilized hollow fiber membrane segments were evaluated under direct immersion liquid phase microextraction sampling mode. Under the optimized experimental conditions, excellent linearity was obtained over the concentration range of 1–250 µgL<sup>-1</sup> for paclobutrazol, and the detection limit was 100 ngL<sup>-1</sup>. The applicability of the present method was validated with real water samples, and the recoveries ranged from 98.6 – 103.1% and relative standard deviations were less than 7.6%. When compared with other reported methods, the presented method is simple, sensitive, inexpensive and efficient technique for the direct determination of paclobutrazol in aqueous samples.

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# EnviroAnalysis 2015 Final Program

Sunday, July 12	Monday, July 13	Tuesday, July 14		Wednesday, July 15	
	Breakfast 7:00 AM – 8:30 AM Vistas Dining Room	Breakfast Sponsored by PerkinElmer 7:00 AM – 8:30 AM: <i>Vistas Dining Room</i>		Breakfast 7:00 AM – 8:30 AM Vistas Dining Room	
	Opening Remarks 8:30 AM – 9:00 AM  Plenary 1. 9:00 AM – 9:45 AM Professor Murray McBride  Plenary 2. 9:45 AM – 10:30 AM Professor Alan Marshall  <i>Room: KC 201</i>	Environmental Impacts of Oil Sands Development <i>Room: KC 201</i>	Spectroscopy <i>Room: KC 205</i> <i>Sponsored by PerkinElmer</i>	Metals in the Environment <i>Room: KC 201</i>	Water <i>Room: KC 205</i>
	Refreshments 10:30 AM – 11:00 AM  <i>Room: KC 203</i>	Refreshments 10:20 AM – 11:00 AM  <i>Room: KC 203</i>		Refreshments 10:20 AM – 11:00 AM  <i>Room: KC 2<sup>ND</sup> Floor Galleria</i>	
	Founders' Award. 11:00 AM – 11:05 AM  Plenary 3. 11:05 AM – 11:50 AM Professor Howard Kingston  <i>Room: KC 201</i>	Plenary 4 11:00 AM – 11:45 AM Professor Jonathan Martin  <i>Room: KC 201</i>		Plenary 5 11:00 AM – 11:45 AM Professor Susan Richardson Sponsored by LECO  <i>Room: KC 201</i>	
	Lunch 11:50 AM – 1:20 PM  <i>Vistas Dining Room</i>	Lunch 11:45 AM – 1:20 PM Sponsored by PerkinElmer <i>Vistas Dining Room</i>		Conference Close 12:00 PM	
	POPs Analysis <i>Room: KC 201</i>	Industrial Applications <i>Room: KC 205</i>	Environmental Impacts of Oil Sands Development <i>Room: KC 201</i>	Mass Spectrometry: Techniques & Applications <i>Room: KC 205</i>	
	Refreshments / Exhibitor Viewing 2:40 PM – 3:40 PM  <i>Room: KC 203</i>		Refreshments / Posters / Exhibitor Viewing 2:40 PM – 3:40 PM  <i>Room: KC 203</i>		
Early Registration 4:30 PM – 5:30 PM  <i>Room: KC 2<sup>ND</sup> Floor Galleria</i>	POPs Analysis <i>Room: KC 201</i>	Industrial Applications <i>Room: KC 205</i>	Environmental Impacts of Oil Sands Development <i>Room: KC 201</i>	Mass Spectrometry: Techniques & Applications <i>Room: KC 205</i>	
	Exhibitor Wine & Cheese Reception Sponsored by Thermo Fisher Scientific 5:00 PM – 6:30 PM <i>Room: KC 203</i>				
Reception 6:00 PM – 9:00 PM <i>Room: KC 105</i>			Exhibit Tear-Down 4:00 PM  Banquet / Karasek Awards Sponsored by Wellington Laboratories 6:00 PM – 10:00 PM: <i>Room: KC 105</i>		