55th Annual

NACRW

NORTH AMERICAN CHEMICAL RESIDUE WORKSHOP

Naples Grande Resort
Naples, Florida
July 22-25, 2018

"Bringing Scientists Together to Develop and Validate Better Methodologies"

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FUTURE MEETING DATES

2019 July 21-24
Naples Grande Resort

2020 July 26-29
Marriott Harbor Beach Resort
Fort Lauderdale

2021 July 25-28
Marriott Harbor Beach Resort
Fort Lauderdale

Dear Attendees, Exhibitors, Sponsors and Guests,

We welcome everyone to the 55th North American Chemical Residue Workshop (NACRW)! We extend a warm greeting to our long-time attendees, our international guests, and our first time participants. We especially thank our Exhibitors and Sponsors for their generous support. Their financial contributions have made it possible for outstanding activities, while maintaining affordable registration fees for attendees. The social events, fantastic technical sessions, and relaxed atmosphere have made NACRW a favorite event for many years!

This year Flag Works, Inc. is dedicating the Conference to Dr. Steven Moser, Oklahoma Department of Agriculture, Food and Forestry, who unexpectedly passed away on June 1, 2018. Steven was a great supporter of NACRW and AOAC International Chemical Contaminants Community regarding pesticide residues. In addition to Chemistry, Steven had many other versatile talents in life. He will be deeply missed!

For those ready to dive into the science upon arrival, we are offering a one-day short course on Sunday. The course, Practical Mass Spectrometry in Residue Chemistry: Basic Principles and Applications, will provide students with a thorough understanding of mass spectrometry as applied to residue chemistry. The instructor, Mike Fillengenzi, California Animal Health and Food Safety Laboratory, University of California at Davis, is a highly experienced residue chemist with many years of practical mass spectrometry experience. We are grateful to have him teach these important concepts to class participants. Also, scheduled on that Sunday afternoon are: Standard Workgroup (Discussion of standard mixes, availability and stability), AOAC International Veterinary Subgroup Meeting, and AOAC International Pesticide Subgroup Meeting.

Our Program Committee has developed a fantastic technical program for you this year. It includes a variety of chemical residue related subjects and special interest topics. As the backbone of the workshop, many aspects of residue analysis will be discussed, including pesticides, veterinary drugs, difficult residues, and matrices. In addition, special topic sessions on natural products, cannabis, emerging contaminants, sample preparation, and high resolution mass spectrometry applications will be featured. We also have the very informative and popular Updates from the Mass Spectrometry Forum.

In addition to our oral sessions please attend the poster sessions, exhibitors, and vendor seminars. The poster authors will be presenting their posters at designated times. This is a great opportunity to engage the authors, ask questions, and cast your all-important vote for best poster. For the second year in a row, NACRW offers student poster awards, sponsored by FLAG Works, Inc. and the ACS Journal of Agricultural and Food Chemistry. The students will be attending the workshop and be available to discuss their work during the allotted time, with the winning student poster announced at the close of the meeting. During the workshop, we encourage attendees to visit our exhibitors to learn more about the products and services they offer for chemical residue testing. We are pleased to offer Vendor seminars starting on Sunday evening and occurring each day of the workshop. This is a great opportunity to hear about the latest developments and discuss your analytical needs with the vendors.

I would like to thank our dedicated volunteers who are on-site working hard to keep the workshop running smoothly. To the 2018 NACRW Organizing Committee, Program Committee, especially Marc Engel and Rob Trengove, Secretary, Katie Carlos, and Executive Director, Teri Besse; it has been a pleasure to work with all of them, and I extend my heartfelt appreciation for all their time and commitment to the workshop. I also want to thank NACRW for this opportunity, it has been a rewarding experience working with everyone.

We hope you enjoy your time at NACRW!

Sincerely,

Alex Krynitsky, 2018 Organizing Committee President
Marc Engel and Rob Trengove, 2018 Program Committee Co-Chairs
2018 Organizing Committee and Program Committee members

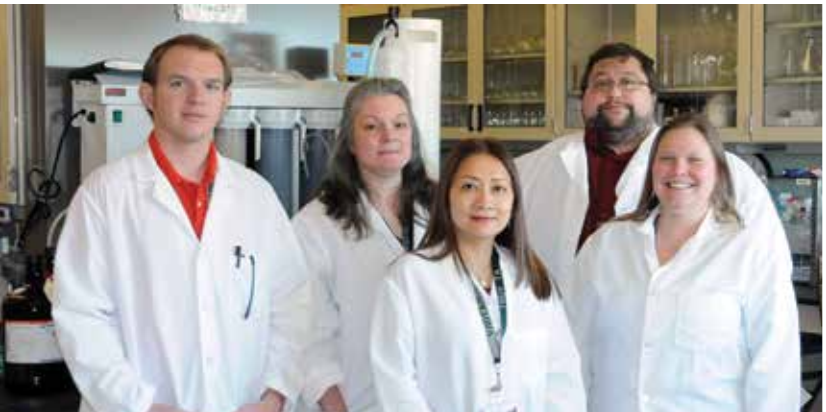
NACRW AND FLAG WORKS, INC. DEDICATES THIS PROGRAM TO THE MEMORY OF STEVEN MOSER

We became better because we met someone special who inspired us. Steven Moser was that special person for many.

A loving father, an achieved scientist trained in mathematics, physics and chemistry, an inventor of multiple sample preparation products, the most kind and giving friend to many, an enthusiastic cook passionate in food chemistry, a weather and space guru, Steven left so many beautiful memories in our hearts. Many friends and colleagues enjoyed hearty laughs with him at conferences and workshops; many say he was so talented in multiple ways; no task was difficult for him; he was always cordial, friendly, helpful and with enthusiasm; he has helped so many agriculture laboratories with their regulatory analysis and has been a tremendous resource and contributor to the community.

Life isn't always fair. The brilliant mind trapped in a body aching for so many years after his post college football career. Steven, may you rest at a good place with all of our prayers and thoughts, without any pain.

We will miss you dearly.



HPLC Methods for N-Methyl Carbamates, Glyphosates and AMPA

USEPA 531.2 | USEPA 547

Pickering Laboratories Offers the Pinnacle PCX and Vector PCX Instruments, SPE and Analytical Columns with Reagents for Carbamate and Glyphosate Analysis in Water and Crops Following the USEPA 531.2 and USEPA 547 Methods.

PICKERING
LABORATORIES

The George and Wilma Fong Founders Award

In Appreciation for Years of Leadership and Dedication to the Florida Pesticide Residue and the North American Chemical Residue Workshop by Volunteering so many hours that contributed to the Advancement of NACRW.

Past Recipients

2011 George and Wilma Fong-Founders

2012 Gail Parker

2013 Pat Beckett

2014 Sherry Garris

2015 Jack Cochran

2016 Amy Brown

2017 Jo Marie Cook

2018 North American Chemical Residue Workshop

FLAG Works, Inc. Board of Directors

Sherry Garris, Chair
Pat Beckett
Jack Cochran
Jo Marie Cook
Susan Eigen
Danny LeCompte
Gail Parker
Sherri Turnipseed
Jon Wong
Teri Besse, Executive Director

Program Committee

Co-Chairs:
Mark Engel, FDACS
Robert Trengove, Murdoch University

Co-Chairs Elect:
Brittany Holmes, WA State Dept. of Agriculture
Ping Wan, Office of Indiana State Chemist

Immediate Past Chair:
Yelena Sapozhnikova, USDA ARS

Program Committee Members

Neely Belai ,US FDA
Willis Chow, Canadian Food Inspection Agency
Brian Eitzer, The Conn. Agr. Exp. Station
Mike Filigenzi, CAHFS, U.C. Davis
Ken Kise, Iowa Dept. of Agriculture
Sara McGrath, US FDA
Steven Moser, ODAFF
Paul Reibach, Smithers Viscient
Sherri Turnipseed, US FDA/ORA/ADRC
Eric Verdon, ANSES
Sam White, US FDA
Jon Wong, US FDA
Paul Yang, Ontario Ministry of the Environment

Poster Committee

Co-Chairs
André de Kok, NVWA
Brittany Holmes, WA State Dept. of Agriculture

Organizing Committee Officers

President:
Alex Krynitsky, Symbiotic Research LLC

Secretary:
Katie Carlos, US FDA CFSAN

President-Elect:
Ken Kise, Iowa Dept. of Agriculture

Immediate Past President:
Kelly Dorweiler, General Mills

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Casandra Benoit, Waters Corporation
Johannes Corley, US FDA
André de Kok, NVWA
Shirley Elliott, Darling Analytical Laboratories
Richard Fussell, Thermo Fisher Scientific
Susie Genualdi, US FDA
Simon Hird, Waters Corporation
Nathan Johnson, Campbell Soup Company
Alaa Kamel, US Environmental Protection Agency
Rebecca Kitlica, FDACS
Joseph Kolb, Merieux Nutrisciences
Joe Konschnik, Restek Corporation
Julie Kowalski, Trace Analytics
Jessica Krank, Colorado Department of Agriculture
Scott Krepich, Phenomenex
Bethany Magrann, UCT
Katerina Mastovska, Covance Laboratories
Cassidy Miller, Agilent Technologies
Allen Misa, Phenomenex
Sareeta Nerkar, Pickering Laboratories
Alexandria Pavkovich, Restek
Lynda Podhorniak,EPA/Office of Pesticide Programs
Michael Riley, GERSTEL
Joe Romano, Waters Corporation
Adam Ross, LGC
Linda Schuchler, Agilent Technologies
Jody Searfoss, UCT
Tameka Taylor, Environmental Protection Agency
Ryan Undeen, Merieux Nutrisciences
Jona Verreth, Montana Dept of Agriculture
Jian Wang, Canadian Food Inspection Agency
Philip Wylie, Agilent Technologies

2018 North American Chemical Residue Workshop Sub-Committee Groups

Communications Committee

Chair: Alexandria Pavkovich
Jody Searfoss
Bethany Magrann
Julie Kowalski

Excellence Award Committee

Chair: Alex Krynitsky
Brad Barrett
Kelly Dorweiler
Ken Kise
Steven Moser
Sareeta Nerkar
Alex Pavkovich
Ping Wan

Short Course Committee

Chair: Brittany Holmes
Jo Marie Cook
Simon Hird
Scott Krepich
Steven Moser
Tameka Taylor
Eric Verdon

Social Event Committee

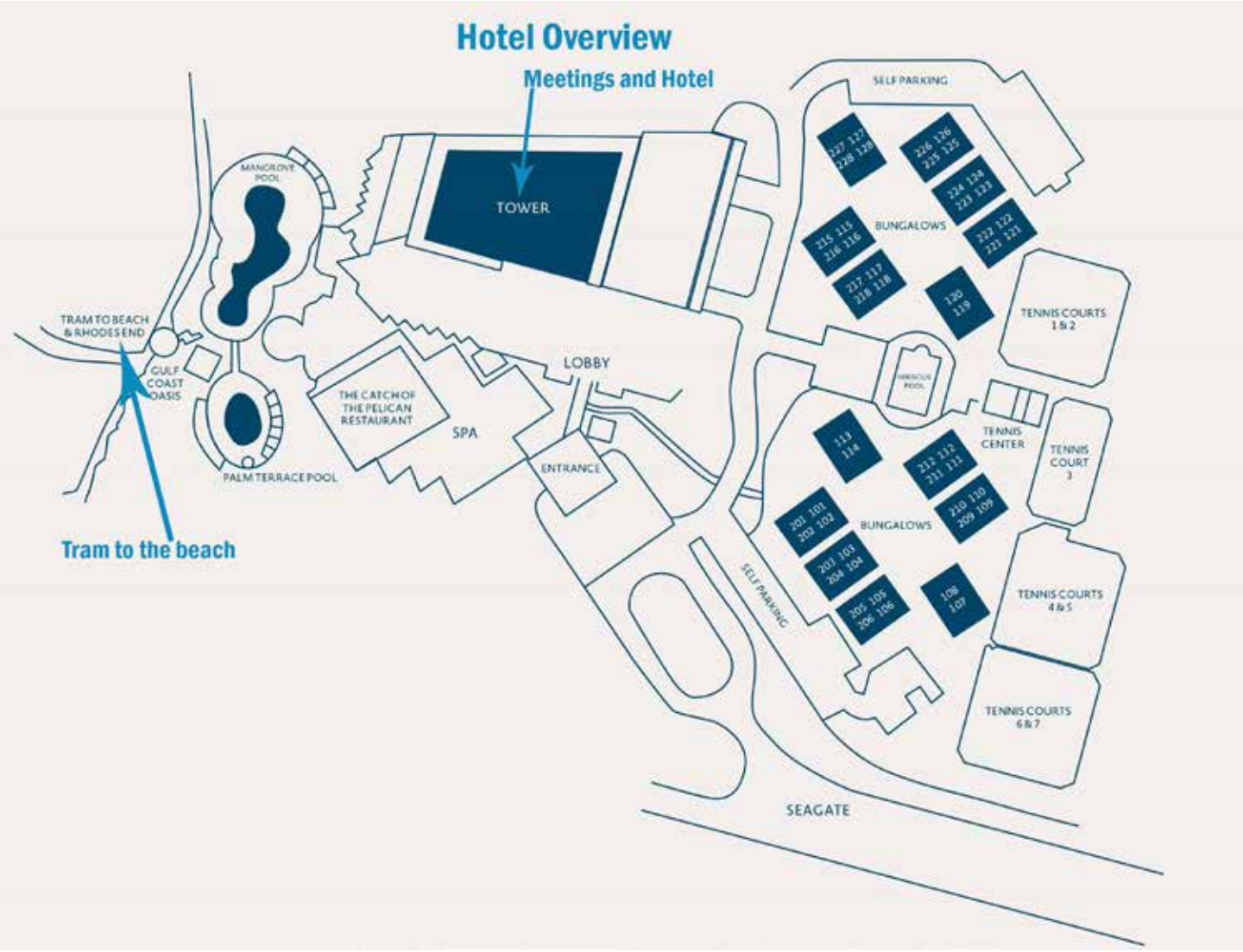
Chair: Sareeta Nerkar
Johannes Corley
Susie Genualdi
Rebecca Kitlica
Joe Konschnik
Julie Kowalski
Steven Moser
Michael Riley

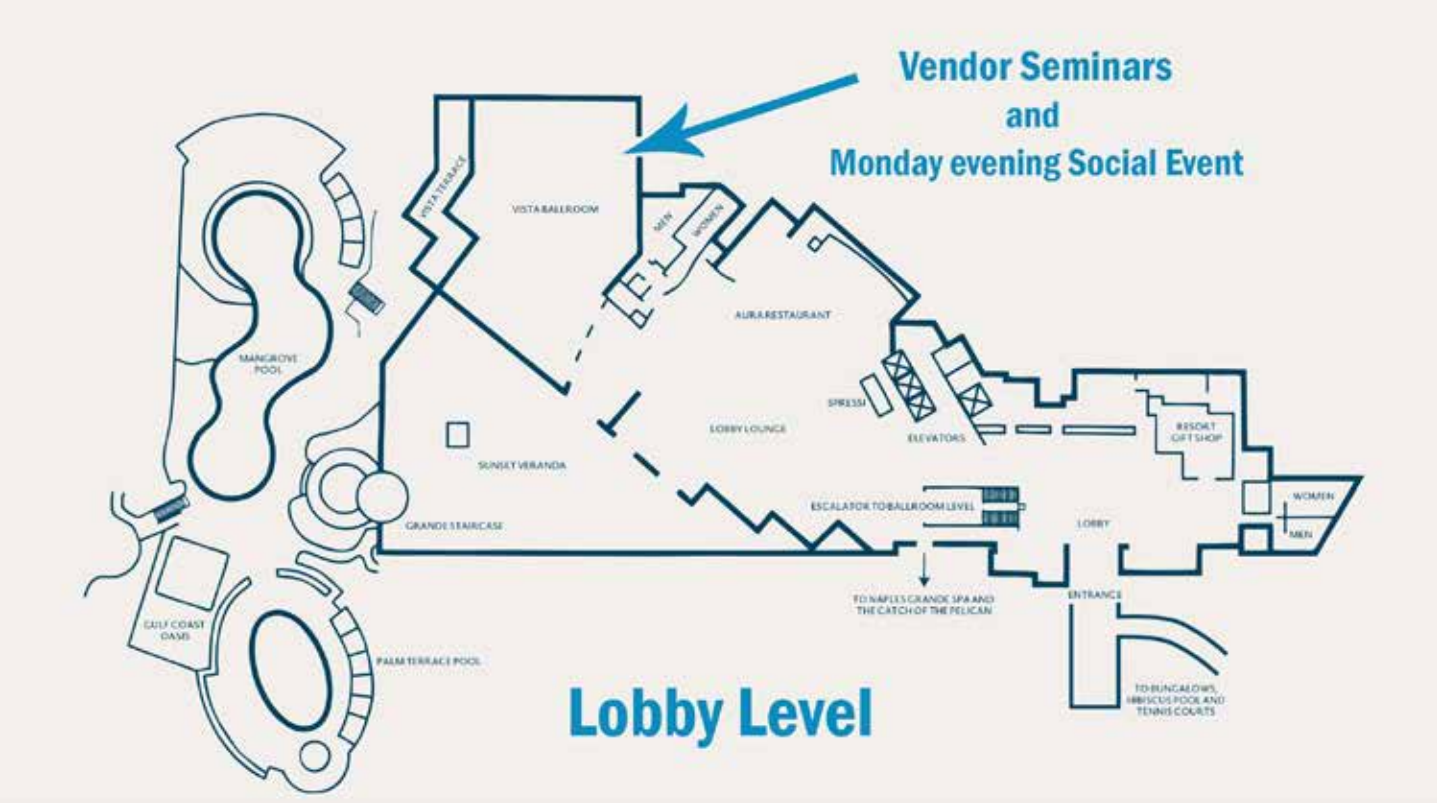
Student Scholarships Committee

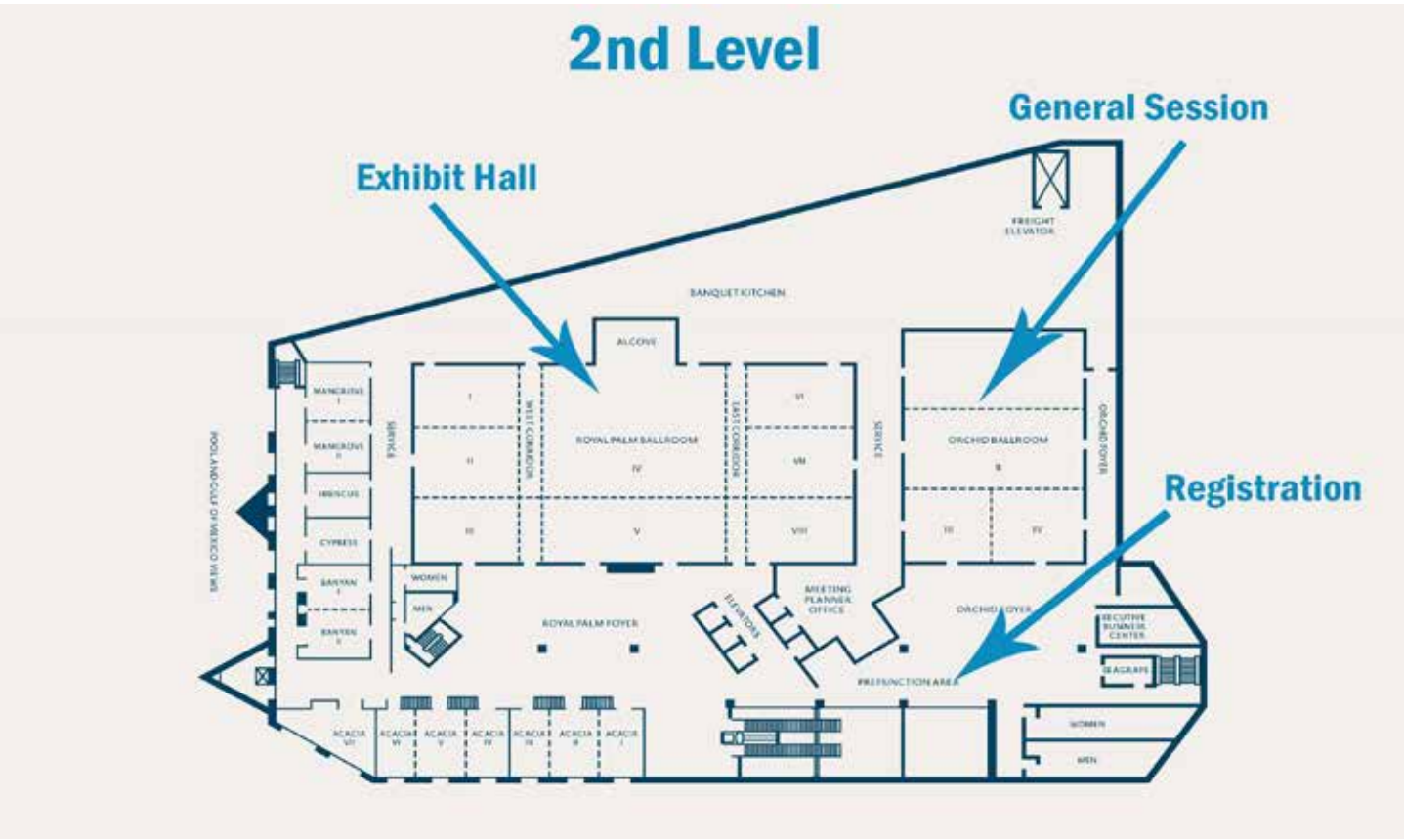
Chair: Katie Carlos
Brian Eitzer
Brittany Holmes
Alaa Kamel
Scott Krepich
Ryan Undeen

to all of our amazing volunteers!

NAPLES GRANDE
BEACH RESORT




Vendor Seminars
and
Monday evening Social Event
VISTA BALLROOM
MEN
WOMEN
AURA RESTAURANT
MANGROVE POOL
SPRESSO
LOBBY LOUNGE
ELEVATORS
RESORT GIFT SHOP
SUNSET VERANDA
ESCALATOR TO BALLROOM LEVEL
WOMEN
MEN
GRANDE STAIRCASE
LOBBY
TO NAPLES GRANDE SPA AND THE CATCH OF THE PELICAN
ENTRANCE
GULF COAST OAKS
PALM TERRACE POOL
TO BUNGALOWS, HIBISCUS POOL AND TENNIS COURTS
Lobby Level


2nd Level
General Session
Exhibit Hall
Registration
FREIGHT ELEVATOR
BANQUET KITCHEN
ALCOVE
ROYAL PALM BALLROOM
ORCHID BALLROOM
MEETING PLANNER OFFICES
ROYAL PALM FOYER
ORCHID FOYER
PRE-FUNCTION AREA
WOMEN
MEN

We would like to thank the following companies for their support of the 2018 NACRW

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BEACH RESORT

MEETING AT A GLANCE

Sunday, July 22, 2018

8:00 am-4:00 pm	**Short Course: Mike Filigenzi** *"Practical Mass Spectrometry in Residue Chemistry: Basic Principles and Applications"*	**Acacia 1-3**
1:00-5:00 pm	Exhibitor Setup	Royal Palm Ballroom
1:00-6:00 pm	Registration	Orchid Foyer
2:00-3:30 pm	**Standards Working Group** *Discussions of standard mixes, availability, and stability*	**Orchid 1-2**
3:45-4:45 pm	**AOAC Veterinary Drugs Subgroup Meeting** *Discussions on AOAC proposal for an collaborative study on multi-residue/multi-class veterinary drug methods; analytes, action levels and target commodities*	**Orchid 1-2**
5:00-6:00 pm	**AOAC Pesticide Subgroup Meeting** *Don't miss out on your opportunity to join your colleagues in discussing pesticide methodology, instrumentation, emerging issues, and so much more. The Pesticide Subgroup family welcomes all new and former attendees of NACRW and looks forward to your valuable contributions.*	**Orchid 1-2**
3:00-6:00 pm	Poster Board Set Up	Royal Palm Ballroom
6:15-7:15 pm	**SCIEX Vendor Seminar**	**Vista Ballroom, 1st level**
7:30-9:30 pm	**Welcome Reception**	**Royal Palm Ballroom**

Monday, July 23, 2018

All Day	Registration	Orchid Foyer
7:00-10:00 am	Poster Board Set Up	Royal Palm Ballroom
7:15-7:45 am	Moderator and Volunteer Training	Orchid Ballroom
7:30-8:15 am	Early Morning Coffee	Orchid Foyer
7:15-8:15 am	**Bruker Vendor Seminar**	**Vista Ballroom, 1st level**
8:30-8:40 am	**Opening Remarks** **Sherry Garris**, Chair, FLAG Works, Inc.	**Orchid Ballroom**
8:40-8:45 am	**Introduction and Presentation of NACRW Excellence Award** **Alex Krynitsky**, 2018 NACRW President	
8:45-9:30 am	**Presentation by Excellence Award Winner** **Janusz Pawliszyn**, Canada Research Chair, Department of Chemistry, University of Waterloo, Waterloo, Ontario, Canada	
9:30-10:45 am	**SESSION 1:** **Sample preparation: new and novel techniques and methods** **Co-Chairs: Steven Moser and Brian Eitzer**	**Orchid Ballroom**
10:45-noon	**Exhibition and Poster Opening**	**Royal Palm Ballroom**
11:00-noon	**Poster Session (authors present for odd #s)**	**Royal Palm Ballroom**
noon-1:00 pm	Cash Lunch (Exhibition Hall)	**Royal Palm Ballroom**
12:15- 1:15 pm	**LECO Corp. Vendor Seminar**	**Vista Ballroom, 1st level**
1:30-3:10 pm	**SESSION 2:** **Advanced and new instrumentation** **Co-Chairs: Willis Chow and Jon Wong**	**Orchid Ballroom**
3:10-3:55 pm	**BREAK (Exhibition & Posters)**	**Royal Palm Ballroom**
3:55-5:35 pm	**SESSION 3:** **Natural products and supplements** **Chair: Ken Kise**	**Orchid Ballroom**
6:30 pm	**Mediterranean Social Event - Naples Grande Beach Resort**	**Sunset Veranda/Vista Ballroom**

MEETING AT A GLANCE

Tuesday July 24, 2018

All Day	Registration	Orchid Foyer
All Day	**Exhibition & Posters**	**Royal Palm Ballroom**
7:30-8:15 am	**Early Morning Coffee**	**Royal Palm Ballroom**
7:00-8:15 am	**Thermo Fisher Scientific Vendor Seminar**	**Vista Ballroom, 1st level**
8:30-10:45 am	**SESSION 4:** **Veterinary Drug Residues** **Co-Chairs: Sherri Turnipseed and Eric Verdon**	**Orchid Ballroom**
10:45-noon	**BREAK (Exhibition & Posters)**	**Royal Palm Ballroom**
11:00-noon	**Poster Session (authors for even #s)**	**Royal Palm Ballroom**
noon-1:00 pm	Cash Lunch (Exhibition Hall)	**Royal Palm Ballroom**
12:15-1:15 pm	**Agilent Technologies Vendor Seminar**	**Vista Ballroom, 1st level**
1:30-3:10 pm	**SESSION 5:** **Natural toxins: fungal and aquatic toxins** **Co-Chairs: Sara Mcgrath and Mike Filigenzi**	**Orchid Ballroom**
3:10-3:55 pm	**BREAK (Exhibition & Posters)**	**Royal Palm Ballroom**
3:55-5:00 pm	**SESSION 6:** **Mass Spectrometry Forum** **Moderators: Brad Barrett and Jack Cochran**	**Orchid Ballroom**
5:05-6:00 pm	**Organization Committee Meeting** *open to all attendees*	**Orchid Ballroom**
6:00-10:30 pm	**Shuttle service to and from Mercato (Blue Martini)**	**Outside - Hotel Main Entrance**

Wednesday, July 25, 2018

Until noon	Registration	**Orchid Foyer**
Until noon	**Exhibition & Posters**	**Royal Palm Ballroom**
7:30-8:15 am	Early Morning Coffee	**Royal Palm Ballroom**
7:15-8:15 am	**Waters Corporation Vendor Seminar**	**Vista Ballroom, 1st level**
8:30-10:30 am	**SESSION 7:** **Environmental and human health** **Co-Chairs: Rob Trengrove and Marc Engel**	**Orchid Ballroom**
10:30-10:45 am	**Student Scholarship Presentations**	**Orchid Ballroom**
10:45-noon	**BREAK (Exhibition & Posters)**	**Royal Palm Ballroom**
12:00-1:00 pm	**o2si smart solutions Vendor Seminar**	**Vista Ballroom, 1st level**
1:05-2:45 pm	**SESSION 8:** **Novel and emerging contaminants of concern in food and the environment** **Co-Chairs: Yelena Sapozhnikova and Paul Yang**	**Orchid Ballroom**
2:45-3:15 pm	**BREAK**	**Orchid Foyer**
3:15-4:55 pm	**SESSION 9:** **Special topics in food and the environment** **Co-Chairs: Neely Belai and Sam White**	**Orchid Ballroom**
4:55-5:10 pm	**Poster Awards and Closing**	**Orchid Ballroom**

Thursday, July 26, 2018

User Meetings

7:30-9:30 am	**SCIEX User Meeting**	**Orchid 2**
7:30-9:30 am	**Thermo Fisher Scientific**	**Orchid 4**
10:30 am – 12:30 pm	**Agilent Technologies**	**Orchid 3**

GENERAL INFORMATION

Registration

Check in once at the registration desk at your earliest opportunity
Sunday - 1:00 – 6:00 pm
Monday - 7:30 am – 5:00 pm
Tuesday - 7:30 am – 5:00 pm
Wednesday - 8:00 am – Noon

KEY to Presentation Numbering System

Oral presentations are numbered O-1, O-2, O-3, O-4, etc.
Vendor Seminars are numbered V-1, V-2, V-3, V-4, etc.
Session A posters are ODD numbered P-1, P-3, P-5, etc.
Session B posters are EVEN numbered P-2, P-4, P-6, etc.

Poster Sessions (Exhibit Hall, Royal Palm Ballroom)

Hang posters Sunday afternoon from 3:00 pm to 6:00 pm or Monday morning from 7:00 am to 10:00 am.
Take down posters between 12 noon to 2:00 pm on Wednesday

Posters may be viewed any time Exhibition is open

Poster Session A (odd#) authors must be at their posters from 11:00 am – noon on Monday and 3:10 - 3:55 pm on Tuesday
Poster Session B (even#) authors must be at their posters from 3:10 pm – 3:55 pm on Monday and 11:00 - noon on Tuesday

Poster Prizes

Two poster prizes of $175 each will be awarded this year, and the same poster/author(s) are eligible to win both prizes. The People's Choice Poster Award will be determined by popular vote of attendees, and the Judges Choice Poster Award will be determined by the poster committee. The criteria used in each case will be importance of the study, quality of the science, and its presentation (including oral discussion and abstract). Also, UCT will present an award for Excellence in Sample Preparation. **Attendees must place their votes in the ballot box by noon on Wednesday.** ***Get a ticket after you turn in your ballot for the chance to win a door prize.***

Exhibition

Sunday evening reception with light hors d'oeuvres and open bar 7:30 pm to 8:30 and cash bar 8:30 pm to 9:30
Monday - 11:00 am - 5:00 pm
Tuesday - 7:30 am – 5:00 pm
Wednesday - 7:30 am – noon

Coffee and Breaks

Coffee will be available 7:30 - 8:00 am on Monday morning in the Orchid Ballroom Foyer and every morning thereafter in the Exhibition Hall (Royal Palm Ballroom). There will also be mid-morning and afternoon refreshment breaks each day. The Monday and Tuesday mid-morning and afternoon breaks, as well as the Wednesday mid-morning break, will be served in the Exhibition Hall (Royal Palm Ballroom). On Wednesday afternoon, the break will be served in the Orchid Ballroom Foyer.

Announcements

Moderators will make general announcements from the podium. If you need to have an announcement made, fill out an announcement form and submit it to Teri Besse or the onsite audio-visual volunteer. These announcement forms will be available at the registration desk.

Job Placement Bulletin Board

Self-serve message board for those offering or seeking employment or to leave notes for others at the meeting.

Door Prizes

Door prizes will be drawn at the end of each morning and afternoon oral session. You must be ON TIME at the beginning of each session to receive a door prize ticket. You must be present at each drawing to win.

Get to Know Your Sponsor

Participate in the "Get to Know Your Sponsor" quiz and win an Apple iPad Pro tablet. A quiz will be provided to you in your registration bag. Simply take the quiz to each sponsor booth, get the right answer and the sponsor will place a sticker on your quiz. After you have completed the quiz, return it to the registration desk no later than Wednesday, July 25th, at 1:30 pm. We will be announcing the winner Wednesday afternoon.

Submission of Manuscripts to *Journal of Agricultural and Food Chemistry*

You are encouraged to contribute original research and/or review articles to the Journal of Agricultural and Food Chemistry for a special section related to the 2018 NACRW. Please inform Robert Trengove, 2018 Program Co-Chair (R.trengove@murdoch.edu.au), by September 7, 2018 if you intend to submit an article. Authors will then be invited by JAFC to submit their manuscripts electronically online through the JAFC website with a deadline of November 30, 2018.

Copies of Presentations

Oral Presentations: Following the meeting, as time and resources permit, oral presentations will be posted on our web site if author permission is granted. There are limitations to what we can post. Absolutely no files will be posted without a speaker's written permission (historically, two thirds of our speakers have given permission). The Power Point files are converted to PDF format, 2 slides per printed page. The file conversion is necessary due to limited server space (the file size of PDF format is roughly 10-20% that of the PPT format). Various security restrictions may be added to the PDF file per speaker's request (such as disabling "copy text" and "print" functions). Some slides containing confidential or proprietary information may be deleted.

Poster Presentations: Drop your business card in the "reprint request" envelope available at each individual poster board. The author should mail/email you a reprint.

Meeting Website

www.NACRW.org - the website includes information on current and future NACRW meetings, as well as archives going back to 2005 and copies of the programs from the start of the workshop!

Meeting Evaluations

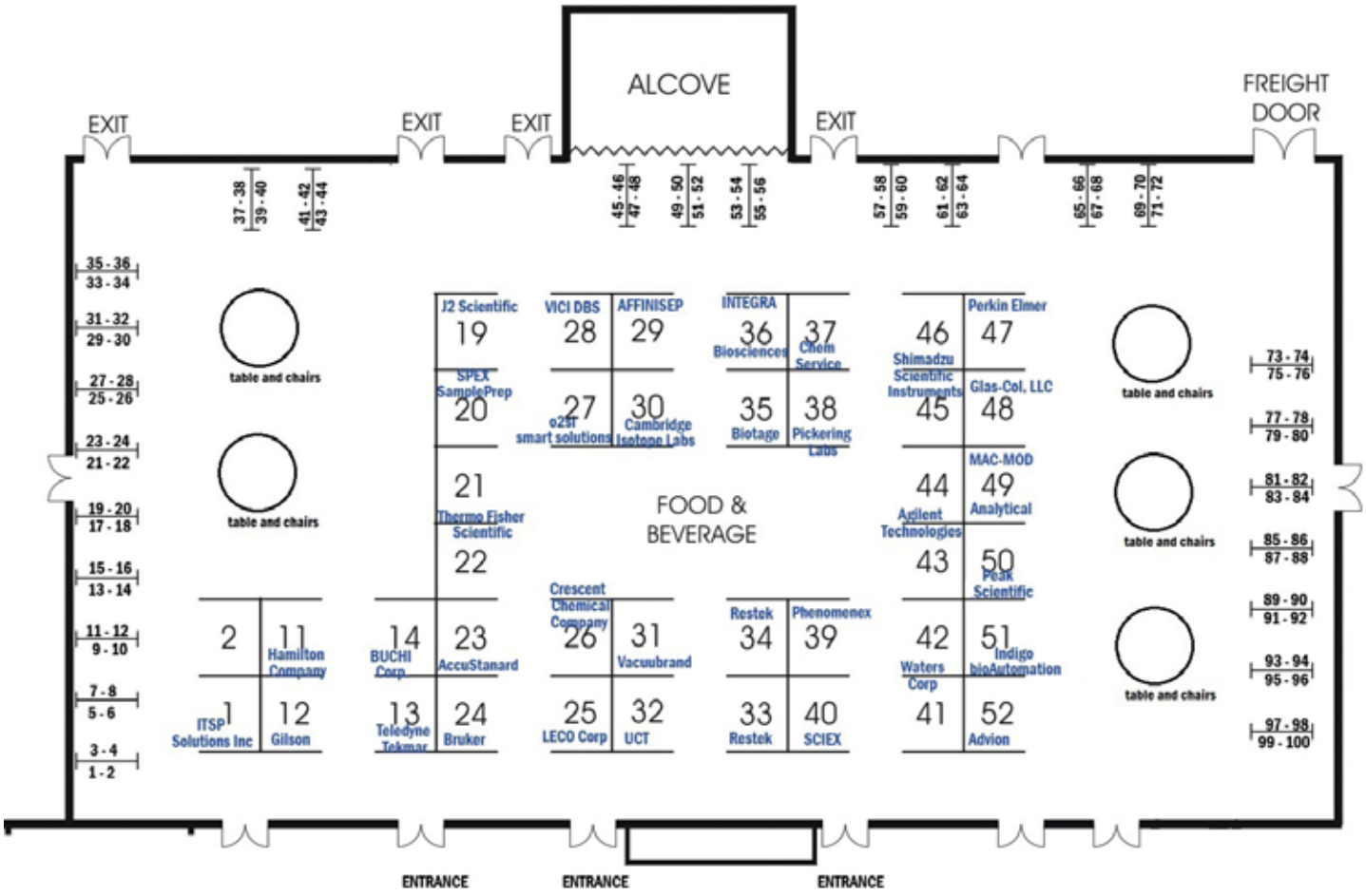
Look for an on-line conference evaluation on the last day of the conference. The evaluation will be emailed to you, so please take a few moments to fill out the online form.

A BIG THANK YOU TO ALL OF OUR VOLUNTEERS, SPONSORS & EXHIBITORS!
The workshop would not be possible without your valuable assistance.

MARK YOUR CALENDAR FOR THE 2019 NACRW
2019 July 21-24 Naples Grande Beach Resort Naples, Florida

Exhibition Hall
and
Poster Sessions

Location: Royal Palm Ballroom, 2nd level



Exhibitors

AccuStandard
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www.accustandard.com

Advion
Booth #52
www.advion.com

AFFINISEP
Booth #29
www.affinisep.com

Agilent Technologies
Booth #43 and 44
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Biotage
Booth #35
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www.indigobio.com

INTEGRA Biosciences
Booth #36
www.integra-biosciences.com/united-states/en

ITSP Solutions Inc
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www.itspsolutions.com

J2 Scientific
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Pickering Laboratories
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Booth #33 and 34
www.restek.com

SCIEX
Booth #40
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Shimadzu Scientific Instruments, Inc.
Booth #45 and 46
www.shimadzu.com

SPEX SamplePrep LLC
Booth #20
www.spexsampleprep.com and www.spexcertiprep.com

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VICI DBS (part of Valco Instruments)
Booth #28
www.vicidbs.com

Waters Corporation
Booth #41 and 42
www.waters.com

SHORT COURSE

Sunday, July 22, 2018 8:00 am to 4:00 pm

Location: Acacia 1-3

PRE-REGISTRATION IS REQUIRED

""Practical Mass Spectrometry in Residue Chemistry: Basic Principles and Applications"

Instructor:

Mike Filigenzi, California Animal Health and Food Safety Lab at UC Davis

This introductory level one-day short course will be focused on the use of mass spectrometry for residue chemistry applications. This course will be aimed towards analysts who are new in the field with the goal being for the students to return to their labs with the kind of knowledge that will immediately allow them to be more productive in their day-to-day work. The course is intended to be interactive with plenty of opportunities for considering questions regarding the various topics as well as discussions of issues students may have encountered in conducting analysis using mass spectrometry. The following topics will be discussed:

- What is a mass spectrometer?
- The history and development of mass spectrometry
- Concepts, terms, and definitions such as resolution, mass accuracy, sensitivity, selectivity, calibration and tuning
- Basic components of a mass spectrometer
- Ion sources – while a number of ion sources will be discussed, the emphasis will be placed on those sources commonly used in residue chemistry: electron ionization and electrospray ionization.
- Mass analyzers, including quadrupole, ion trap, TOF, and orbitrap systems
- Hybrid mass analyzers, including quadrupole-ion trap and quadrupole-TOF systems
- Qualitative identification considerations including the use of electron ionization MS libraries and requirements for positive identification by various techniques.
- Quantitative considerations including terms and definitions related to quantitative methods such as precision, accuracy, and linearity and how these are measured. The use of isotopically labeled analogs and the effects of sample matrices on instrument response will also be discussed.
- Various applications of MS including highly targeted, low level analysis, broad range screening, and analysis for unknown contaminants using different types of systems.

Analytical Standards and Sample Preparation Equipment for Chemical Residue Testing

Visit SPEX CertiPrep and SPEX SamplePrep at NACRW, Booth #20

SPEX CertiPrep provides superior Certified Reference Materials for food testing and has been serving the scientific community since 1954.

Pesticide Mixes and Kit

We have designed a **Pesticide Residue Testing Kit** which includes 144 of the most commonly analyzed pesticides per EPA, AOAC, FDA and other international testing methods. Shorter calibration times, fewer injections and money savings, as compared to purchasing individual pesticide standards.

Testing Kits

Our **Heavy Metals and Minerals Testing Kits** are designed for routinely analyzed heavy metals and minerals. All kits come with six, 30 mL ICP-MS standards which includes a nitric acid blank for easy dilution.

Poster Presentation

View our poster, **"Not Your Kid's Apple Juice: An Examination of Arsenic Content in American and European Hard Ciders"**. Our poster will be on display and available for viewing in the Royal Palm Ballroom of the Naples Grande Hotel, Monday, July 23, at 11 a.m., until Wednesday, July 25, at noon.

For over 60 years, SPEX SamplePrep has been providing solutions for difficult and unique sample preparation problems for the agricultural industry worldwide.

Tools for QuEChERS

The **2010 Geno/Grinder®** provides high throughput for QuEChERS. It is designed for vigorous up-and-down shaking of up to twenty-four 15 mL tubes or sixteen 50 mL centrifuge tubes simultaneously.

The **1600 MiniG®** is useful for labs that process fewer samples for QuEChERS. It agitates up to six 50 mL or 15 mL vials.

Grinders for Tough Samples

The **6875 Freezer/Mill®** is a cryogenic grinder that is able to pulverize the toughest samples while preserving temperature sensitive compounds.

These products can process plant and animal tissue, as well as vegetables and fruit. They offer improved consistency and extraction efficiency compared to hand shaking alone. Contact us today to find out how you can get a free demonstration in your lab.

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VENDOR SEMINARS

Food and beverage provided by each company

(PRE-REGISTRATION IS REQUIRED)
Please sign up at the meeting registration desk

V-1 Sunday Evening, July 22, 2018, 6:15 p.m. to 7:15 p.m. SCIEX
Location: Vista Ballroom

DuoSpray™ Ionization: A Novel Approach to Analyzing the California Mandated List of Pesticides in Cannabis

KC Hyland, Global Technical Marketing Mgr – Food and Environmental

Legalization of *Cannabis* in the United States is defined by individual states with varying legislation for pesticides to be monitored and at what levels. Legislation for pesticides residue testing usually contains action limits which residues are not allowed to exceed. The California List consists of two categories and expands on Oregon's pre-existing suite of pesticides by adding six additional compounds and generally lowering pesticide action limits relative to Oregon's limits. The panel requires electrospray ionization (ESI) and atmospheric pressure chemical ionization (APCI) to be analyzed in its entirety by LC-MS/MS. The SCIEX DuoSpray™ Ion Source consists of two probe housing components, for ESI and APCI, and can be operated on the front of the SCIEX QTRAP® 6500+ system, providing flow to either probe for controlling the ionization over the chromatographic gradient. Analysis of the CA List in a single injection using positive/negative polarity switching and both ionization techniques is enabled in this manner. Pesticides historically analyzed by gas chromatography can now be captured in a single LC-MS/MS injection as part of a more comprehensive residues panel. Data for all 66 pesticides on the California list in Cannabis flower extract show that Category II action limits were achieved for inhalable products when coupled to a QTRAP® 6500+ system.

V-2 Monday, July 23, 2018, 7:15 to 8:15 am BRUKER
Location: Vista Ballroom

Full quantitative and qualitative validation of a LC-Q-TOF multiresidue method for 272 pesticides in food samples

André de Kok and Jos Scholten
NVWA - Netherlands Food and Consumer Product Safety Authority, NRL for Pesticide Residues in Food and Feed, Akkermaalsbos 4, 6708WB Wageningen, The Netherlands; a.dekok@nvwa.nl

High-Resolution Accurate Mass (HRAM) Mass Spectrometry, based on e.g. Time-of-Flight (TOF) technology, has considerably improved the possibilities of multiresidue methods, particularly in terms of unlimited-scope of analytes and highest possible level of identification confidence.
In this presentation, the full validation of a LC-Q-TOF multiresidue method for 272 pesticides in food products using the NL-method (lettuce and orange) and QuEChERS-method (chicken meat) for extraction (and cleanup), respectively, will be presented. The fulfilment of the EU AQC SANTE Document validation requirements will be shown. This will also been done for a comparison study for the detection and identification of 155 representative pesticides in 20 different matrices.

Finally, a critical comparison will be shown of our UHPLC-Q-TOF and high-end UHPLC-MS/MS TQ instruments, as to sensitivity, selectivity and requirements for data processing software, and the influence on ease of use for routine application of HRMS-methods will be highlighted.

V-3 Monday, July 23, 2018, 12:15 to 1:15 pm **LECO Corporation**
Location: Vista Ballroom

Comprehensive, Non-Target Characterization of Environmental Exposome Samples Using GCxGC & High Resolution Time-of-Flight Mass Spectrometry

Lorne M. Fell, Joseph E. Binkley, Todd S. Richards, LECO –Separation Sciences Applications Centre

Historically targeted analysis has been the primary route to evaluate complex environmental samples. This constrained testing, while effective, has often missed emerging or unexpected compounds within samples. Recent improvements in detection and data processing capabilities of various systems have allowed scientists to more fully evaluate these same samples using non-targeted (NT) techniques. As a result, the EPA is currently conducting a multiple lab, multiple platform evaluation for non-targeted analysis methods in samples designed to mimic the environmental exposome. The project contains two initial phases, first a blinded study is conducted and reported. In phase two the individual standard component lists are provided and the evaluation revised as necessary. Each blind standard is reported to contain between 100-400 spiked analytes with potential for more due to contaminants, intra-sample degradation, or reaction product formation. This presentation describes the systematic logic used for identification of the unknowns, its results, and the lessons learned from the process as it applied to the first round of ten, blinded ENTACT samples for a single platform. The platform used was a comprehensive GCxGC gas chromatograph coupled with a high resolution accurate mass (HRAM) time-of-flight mass spectrometer (TOF-MS) in both electron ionization (EI) and chemical ionization (CI) modes. Deconvolved spectra were matched to existing commercial MS libraries and screened based on the peak's retention index value, molecular ion mass accuracy, and fragment ion formula fidelity. Questions addressed will be : (i) what percentage of each sample was correctly identified, (ii) what instrumentation characteristics contributed most significantly to the identification, and (iii) what impurities, reaction products and degradation products were identified.

V-4 Tuesday, July 24, 2018 7:00 to 8:15 am **THERMO FISHER SCIENTIFIC**
Location: Vista Ballroom

Targeted and Non-Targeted MS Methods for Algal Toxins and their Potential Health Effects

At NACRW 2018, Thermo Fisher Scientific is pleased to have two highly respected researchers present their work on toxins produced by cyanobacteria (blue-green algae) blooms and potential health outcomes related to exposure to those toxins.

Dr. Judy Westrick from Wayne State University will provide an overview of algal toxins along with the analytical methods that her laboratory employs for both targeted and non-targeted analysis including LC-MS-MS, LC-IT-HRMS and ELISA.

Dr. David Muddiman from North Carolina State University will discuss research his lab has undertaken that looks at exposure to cyanotoxins in the food web and the possible relationship that exposure has to sporadic amyotrophic lateral sclerosis (ALS).

V-5 Tuesday, July 24, 2018, 12:15 to 1:15 pm **AGILENT TECHNOLOGIES**
Location: Vista Ballroom

Application of Enhanced Matrix Removal-Lipid Cartridges for Sample Preparation in Food Analysis

Limian Zhao, Senior SPP Application Scientist.
Agilent Technologies, Wilmington, DE USA 19808

Captiva Enhanced Matrix Removal-Lipid (EMR-Lipid) implements a pass-through solid phase extraction (SPE) format for highly selective lipid removal without impacting analyte recovery. Lipid hydrocarbon chains are trapped in the sorbent while analytes pass-through for analysis using a combined mechanism of size exclusion and hydrophobic interaction. The pass-through cleanup is faster and easier than traditional fat cleanup methods and provides high lipid removal efficiency. The cleaner samples improve method reliability and data quality, reduce negative matrix impact on the instrument, and improves the sample analysis productivity.

Trim time not columns – Multiresidue Pesticide analysis with the Intuvo GC/TQ

Melissa Churley, GCMS Applications Scientist
Agilent Technologies, Santa Clara, CA USA 95051

The Intuvo 9000GC system provides an entirely new approach to optimize productivity in the GCMS laboratory with innovations such as a compact, no-trim column and easy-to-install, disposable Guard Chip. A new and robust method, coupling the Intuvo 9000GC with a 7000D GC TQ MS system, will be described as we introduce the new Agilent Pesticide and Environmental Pollutants MRM Database and Workflow Kit for the Intuvo GC/TQ. The kit includes a customized retention-time-locked MRM database designed specifically for the Intuvo GC, which includes matrix-matched transitions and retention time information for the multiresidue screening of pesticides in food and easy reference to other consumables needed to stay productive.

V-6 Wednesday, July 25, 2018, 7:15 to 8:15 am **WATERS CORPORATION**
Location: Vista Ballroom

Part 1:
What's New and Exciting in 2018 from Waters!

Simon Hird, Principle Scientist, Waters Corporation, Wilmslow, UK

Sample preparation, separation, MS detection, and informatics; it's all important! This presentation will provide an insight into what is new in each of these critical technology areas from Waters in 2018.

Part 2:
Ion Mobility-HRMS Based Screening of Suspected Compounds in Complex Matrices without the Use of Physical Reference Substances

Anton Kaufmann, Official Food Control Authority Zürich, Switzerland

Modern multiresidue methods analyse several hundreds of compounds within a single chromatographic run (e.g. pesticides in fruits). Such methods rely on the use of physical reference compounds in order to confirm suspected peaks. It is very challenging to produce and maintain reference compound solutions containing hundreds of anaytes. Hence, there is a strong motivation to move away from relying on physical reference substances.

Data independent acquisition (DIA) can be used to produce product ion information for each observed chromatographic peak in a complex sample. Such product ion data can be used to tentatively confirm suspected analytes by in-silico fragmentation data.

The DIA approach investigated in this presentation was based on ion mobility Q-TOF technology (Vion). This instrument monitors a comprehensive 4-dimensional space consisting of retention time, drift time, m/z and ion abundance. A “chopping” in-silico fragmentation algorithm (MassFragment) is fed with the chemical structures of the targeted compounds (mol file). Each chromatographic peak is tested for the presence of the accurate mass of the precursor and any of the accurate masses of the postulated product ions.

This approach produced encouraging results even when analysing residues in complex matrices (e.g. veterinary drugs in bovine liver extracts).

V-7 Wednesday, July 25, 2018, 12:00 to 1:00 pm **O2SI SMART SOLUTIONS**
Location: Vista Ballroom

The Challenge of Designing a Master Calibration Solution for Pesticide Solution Product Verification

Huichen Stavros, Ph.D. and Daniel Biggerstaff, Ph.D.

As a custom reference material manufacturer, we produce approximately 400 solutions weekly with about 25% of those solutions being made for the first time. This places a high demand on generating excellent data quality and high throughput in the quality control laboratory. To facilitate both of these aspects, we have developed GC/MS and LC/HRAMS pesticide calibration method containing approximately 400 and 800 analytes, respectively. Stability data, solvent, pH, and chemistries will be discussed.

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2018 NACRW EXCELLENCE AWARD

PRESENTED TO

Dr. Janusz Pawliszyn

B.Sc./Chem.Eng., 1977, Technical University of Gdansk
M.Sc., 1978, Technical University of Gdansk
Ph.D., 1982, Southern Illinois University
PDF., 1984, University of Toronto

The primary focus of Professor Pawliszyn's research program is the design of highly automated and integrated instrumentation for the isolation of analytes from complex matrices and the subsequent separation, identification and determination of these species. The primary separation tools used by his group are Gas Chromatography, Liquid Chromatography and Capillary Electrophoresis coupled to variety of detections systems, including range of mass spectrometry techniques. Currently his research is focusing on elimination of organic solvents from the sample preparation step to facilitate on-site monitoring and in-vivo analysis. Several alternative techniques to solvent extraction are investigated including use of coated fibers, packed needles, membranes and supercritical fluids. Dr. Pawliszyn is exploring application of the computational and modeling techniques to enhance performance of sample preparation, chromatographic separations and detection. The major area of his interest involves the development and application of imaging detection techniques for microcolumn chromatography, capillary electrophoresis and micro chip separation devices.

Professor Pawliszyn has supervised 45 PhD and 64 MS students and he is an author of over 550 scientific publications and a book on Solid Phase Microextraction. His Hirsch Index (H-index) is 88. He is a Fellow of Royal Society of Canada and Chemical Institute of Canada, editor of Analytica Chimica Acta, Trends in Analytical Chemistry and a member of the Editorial Board of Journal of Separation Science and Journal of Pharmaceutical Analysis. He initiated a conference, "ExTech", focusing on new advances in sample preparation and disseminates new scientific developments in the area, which meets every year in different part of the world. He received the 1995 McBryde Medal, the 1996 Tswett Medal, the 1996 Hyphenated Techniques in Chromatography Award, the 1996 Caledon Award, the Jubilee Medal 1998 from the Chromatographic Society, U.K., the 2000 Maxxam Award from Canadian Society for Chemistry, the 2000 Varian Lecture Award from Carleton University, the Alumni Achievement Award for 2000 from Southern Illinois University, the Humboldt Research Award for 2001, 2002 COLACRO Medal, 2003 Canada Research Chair, in 2006 he has been elected to the most cited chemists by ISI, in 2008 he received A.A. Benedetti-Pichler Award from Eastern Analytical Symposium, 2008 Andrzej Waksmundzki Medal from Polish Academy of Sciences, 2008 Manning Principal Award, 2010 Torbern Bergman Medal from the Swedish Chemical Society, 2010 Ontario Premier's Innovation Award, 2010 Marcel Golay Award, 2010 ACS Award in Separation Science and Technology, 2011 PittCon Dal Nogare Award, 2012 E.W.R. Steacie Award, 2013 CIC Environmental Research and Development Award, 2013 CIC LeSueur Memorial Award, 2015 Maria Skłodowska-Curie Medal from Polish Chemical Society, 2015 Halász Medal Award from the Hungarian Society for Separation Sciences, 2017 Pittsburgh Conference Analytical Chemistry Award, the 2017 Eastern Analytical Symposium Award for Outstanding Achievements in the Fields of Analytical Chemistry and 2018 ACS Award in Chromatography. He presently holds the University Professor, Canada Research Chair and Natural Sciences and Engineering Research Council of Canada Industrial Research Chair in New Analytical Methods and Technologies.

The title of Dr. Pawliszyn's presentation is:

"Matrix Compatible Coatings in SPME: An Enabling Technology Facilitating Full Automation and On-site Residue Determinations of Food Products"

MEETING PROGRAM

Sunday, July 23, 2017

8:00 am-4:00 pm	**Short Course: Mike Filigenzi** ***"Practical Mass Spectrometry in Residue Chemistry: Basic Principles and Applications"***	**Acacia 1-3**
1:00-5:00 pm	Exhibitor Setup	Royal Palm Ballroom
1:00-6:00 pm	Registration	Orchid Foyer
2:00-3:30 pm	**Standards Working Group** *Discussions of standard mixes, availability, and stability*	**Orchid 1-2**
3:45-4:45 pm	**AOAC Veterinary Drugs Subgroup Meeting** *Discussions on AOAC proposal for a collaborative study on multi-residue/multi-class veterinary drug methods; analytes, action levels and target commodities*	**Orchid 1-2**
5:00-6:00 pm	**AOAC Pesticide Subgroup Meeting** *Don't miss out on your opportunity to join your colleagues in discussing pesticide methodology, instrumentation, emerging issues, and so much more. The Pesticide Subgroup family welcomes all new and former attendees of NACRW and looks forward to your valuable contributions.*	**Orchid 1-2**
3:00-6:00 pm	Poster Board Set Up	Royal Palm Ballroom
6:15-7:15 pm	**SCIEX Vendor Seminar**	**Vista Ballroom, 1st level**
7:30-9:30 pm	**Welcome Reception**	**Royal Palm Ballroom**

Monday, July 23, 2018

All Day	Registration	Orchid Foyer
7:00-10:00 am	Poster Board Set Up	Royal Palm Ballroom
7:15-7:45 am	Moderator and Volunteer Training	Orchid Ballroom
7:15-8:15 am	**Bruker Vendor Seminar**	**Vista Ballroom, 1st level**
7:30-8:15 am	Early Morning Coffee	Orchid Foyer
8:30-8:40 am	**Opening Remarks** **Sherry Garris**, Chair, FLAG Works, Inc.	**Orchid Ballroom**
8:40-8:45 am	**NACRW Excellence Award Presentation and Keynote Address** **Introduction and Presentation of NACRW Excellence Award** **Alex Krynitsky**, 2018 NACRW President	
8:45-9:30 am	**Presentation by Excellence Award Winner** **Janusz Pawliszyn**, Canada Research Chair, Department of Chemistry, University of Waterloo, Waterloo, Ontario, Canada	
A-1	**Matrix Compatible Coatings in SPME: An Enabling Technology Facilitating Full Automation and On-site Residue Determinations of Food Products**	

9:30-10:45 am	**SESSION 1:** **Sample preparation: new and novel techniques and methods** **Co-Chairs: Brian Eitzer**	**Orchid Ballroom**
9:30-9:50 am **O-1**	**Sonia Herrera López**, NVWA- Netherlands Food and Consumer Product Safety Authority, Wagenningen, Netherlands **Comprehensive study of different approaches for the determination of 15 highly polar pesticides in food matrices. Influence of matrix type on the identification**	
9:55-10:15 am **O-2**	**Michael S Young**, Waters Corporation, Milford, MA, USA **Improved Extraction and Cleanup Prior to LC-MS/MS Determination of Bound and Unbound Dicamba and Dicamba Metabolite Residues in Agricultural Samples**	
10:20-10:40 am **O-3**	**Shabarinath Nambiar**, Murdoch University, Perth, WA, Australia **Single tube extraction and clean up using solubilized QuEChERS extractants – a miniaturized approach**	
10:45-noon	**Exhibition and Poster Opening**	**Royal Palm Ballroom**
11:00-noon	**Poster Session (authors present for odd #s)**	**Royal Palm Ballroom**
noon-1:00 pm	Cash Lunch (Exhibition Hall)	**Royal Palm Ballroom**
12:15- 1:15 pm	**LECO Corp. Vendor Seminar**	**Vista Ballroom, 1st level**
1:30-3:10 pm	**SESSION 2:** **Advanced and new instrumentation** **Co-Chairs: Willis Chow and Jon Wong**	**Orchid Ballroom**
1:30-1:50 pm **O-4**	**Lili He**, University of Massachusetts, Amherst, MA, USA **Surface Enhanced Raman Spectroscopy for Food Colorant Analysis**	
1:55-2:15 pm **O-5**	**Scott White**, University of Minnesota, Minneapolis, MN, USA **Food Sensing with Printed, Floating-Gate Transistors**	
2:20-2:40 pm **O-6**	**Kaveh Jorabchi**, Georgetown University, Washington, DC, USA **Plasma Assisted Reaction Chemical Ionization: A Sensitive Ion Source for Elemental Detection and Quantification of Residues**	
2:45-3:05 pm **O-7**	**Amadeo R. Fernández-Alba**, European Union Reference Laboratory for Pesticide Residues in Fruit & Vegetables, University of Almeria, Almería, Spain **Pesticides analysis in complex matrices by new GC and LC MS/MS improved platforms**	
3:10-3:55 pm	**BREAK (Exhibition & Posters)**	**Royal Palm Ballroom**
3:55-5:35 pm	**SESSION 3:** **Natural products and supplements** **Chair: Ken Kise**	**Orchid Ballroom**
3:55-4:15 pm **O-8**	**Todd Richards**, LECO, Saint Joseph, MI, USA **Utilizing GCxGC-TOFMS for Quantitation of Cannabis Related Pesticides in Hops QuEChERS Extract**	
4:20-4:40 pm **O-9**	**Wesley Maguire**, Pixis Labs/Columbia Food Lab, Portland, OR, USA **Comprehensive Determination of Unregulated Pesticides in Cannabis Flower using Tandem Mass Spectrometry**	

4:45-5:05 pm **Avinash Dalmia**, PerkinElmer, Shelton, CT, USA
O-10 **Analysis of all pesticide residues in cannabis regulated by California and Oregon state using LC/MS/MS with Electrospray and APCI source**

5:10-5:35 pm **Agustin Pierri,** Weck Laboratories, Industry, CA, USA
O-11 **Cannabis Sativa pesticides, aflatoxins, and potency by LC/MS/MS: one extraction, one analysis**

6:30 pm **Mediterranean Social Event - Naples Grande** **Sunset Veranda/Vista Ballroom**

Tuesday July 24, 2018

All Day Registration
All Day **Exhibition & Posters** **Royal Palm Ballroom**

7:00-8:15 am **Thermo Fisher Scientific Vendor Seminar** **Vista Ballroom, 1st level**

7:30-8:15 am **Early Morning Coffee** **Royal Palm Ballroom**

9:00-10:45 am **SESSION 4:** **Orchid Ballroom**
Veterinary drug residues
Co-Chairs: Sherri Turnipseed and Eric Verdon

9:00-9:20 am **Kevin C. Hsieh**, Florida Department of Agriculture and Consumer Services, Tallahassee, FL, USA
O-12 **Accurate Quantitation and Analysis of Nitrofurans and Phenicols in Seafood by UHPLC-MS/MS: Proficiency Testing and State Regulatory Samples**

9:25-9:45 am **Ovokeroye A. Abafe**, Agricultural Research Council-OVR, Onderstepoort, South Africa
O-13 **Development and validation of a confirmatory method for the determination of stilbene estrogens in Ostrich serum**

9:50-10:10 am **Anton Kaufmann**, Official food control authority of the Canton of Zurich, Zurich, Switzerland
O-14 **Moving from targeted towards non-targeted approaches**

10:15-10:40 am **Thierry Delatour**, Nestlé Research Centre, Lausanne, Switzerland
O-15 **Matrix effect-monitored screening of a hundred veterinary drug residues in food by liquid chromatography-triple quadrupole mass spectrometry**

10:45-noon **BREAK (Exhibition & Posters)** **Royal Palm Ballroom**
11:00-noon **Poster Session B (authors for even #s)** **Royal Palm Ballroom**
noon-1:00 pm Cash Lunch (Exhibition Hall) **Royal Palm Ballroom**

12:15-1:15 pm **Agilent Technologies Vendor Seminar** **Vista Ballroom, 1st level**

1:30-3:10 pm **SESSION 5:** **Orchid Ballroom**
Natural toxins: fungal and aquatic toxins
Co-Chairs: Sara Mcgrath and Mike Filigenzi

1:30-1:50 pm **Sareeta Nerkar**, Pickering Laboratories Inc., Mountain View, CA, USA
O-16 **HPLC Method for Quantification of Mycotoxins in Animal Matrices, Food and Feed**

1:55-2:15 pm **Geoffrey Faden**, Mac-Mod Analytical, Chadds Ford, PA, USA
O-17 **Fast, highly sensitive analysis of mycotoxins using superficially porous particles**

2:20-2:40 pm **O-18**	**Wendy K. Strangman**, University of North Carolina Wilmington, Wilmington, NC, USA **What are we missing? Metabolomics, toxin analysis, and new compound discovery from marine and freshwater HAB species**	
2:45-3:05 pm **O-19**	**Pearse McCarron**, National Research Council Canada, Halifax, NS, Canada **Analytical Methods and Reference Materials for Marine Toxins**	
3:10-3:55 pm	**BREAK (Exhibition & Posters)**	**Royal Palm Ballroom**
3:55-5:00 pm	**SESSION 6:** **Mass Spectrometry Forum** **Moderators: Brad Barrett and Jack Cochran**	**Orchid Ballroom**
5:05-6:00 pm	**NACRW Organizing Committee Meeting** *open to all attendees*	**Orchid Ballroom**
6:00-10:30 pm	**Shuttle service to and from Mercato (Blue Martini)** *One shuttle scheduled to run every 15 minutes*	**Outside - Hotel Main Entrance**

Wednesday, July 25, 2018

Until noon	Registration	**Orchid Foyer**
Until noon	**Exhibition & Posters**	**Royal Palm Ballroom**
7:15-8:15 am	**Waters Corporation Vendor Seminar**	**Vista Ballroom, 1st level**
7:30-8:15 am	Early Morning Coffee	**Royal Palm Ballroom**
8:30-10:45 am	**SESSION 7:** **Environmental and human health** **Co-Chairs: Rob Trengove and Marc Engel**	**Orchid Ballroom**
8:30-8:55 am **O-20**	**Zachary Laughrey**, USGS, Lawrence, KS, USA **Analysis of Cyanotoxin and Algal Toxins: Data gaps, Challenges, and Advancements**	
9:00-9:25 am **O-21**	**Anthony Macherone**, Agilent Techonologies, Wilmington, DE, USA **Integrating GC-TOF exposome profiling and genetic disease screening to provide a holistic perspective on honey bee health**	
9:30-9:55 am **O-22**	**P. Barry Ryan**, Emory University, Atlanta, GA, USA **Insecticide Exposures and Human Health**	
10:00-10:25 am **O-23**	**Danielle Stanek**, Florida Department of Health, Tallahassee, FL, USA **One Health Approach to Chemical Residues in Our Environment**	
10:30-10:45 am	**Student Scholarship Presentations**	**Orchid Ballroom**
10:45-noon	**BREAK (Exhibition & Posters)**	**Royal Palm Ballroom**
12:00-1:00 pm	**o2si smart solutions Vendor Seminar**	**Vista Ballroom, 1st level**

1:05-2:45 pm **SESSION 8:** **Orchid Ballroom**
Novel and emerging contaminants of concern in food and the environment
Co-Chairs: Yelena Sapozhnikova and Paul Yang

1:05-1:25 pm **Katerina Mastovska**, Covance Laboratories, Madison, WI, USA
O-24 **Reliable and Cost-Effective Analysis of Bisphenol A and Nonylphenols in Foods and Beverages**

1:30-1:50 pm **Yelena Sapozhnikova**, USDA, Wyndmoor, PA, USA
O-25 **Analysis of organophosphate esters in meats and fish**

1:55-2:15 pm **Hui Peng,** University of Toronto, Toronto, Canada
O-26 **Untargeted Screening and Prioritization of Halogenated Chemicals in the Environment**

2:20-2:40 pm **Mark W Sumarah,** Agriculture and Agri-food Canada, London, Canada
O-27 **Non-targeted Analysis of Emerging Environmental Contaminants by Data Independent Acquisition LC-MS/MS**

2:45-3:15 pm **BREAK** **Orchid Foyer**

3:15-4:55 pm **SESSION 9:** **Orchid Ballroom**
Special topics in food and the environment
Co-Chairs: Neely Belai and Sam White

3:15-3:35 pm **Sitra Abubeker**, USDA Agricultural Marketing Service, Washington, DC, USA
O-28 **USDA's Pesticide Data Program-Who Uses the Data**

3:40-4:00 pm **Barbara Kiedrowska**, NVWA - Netherlands Food and Consumer Product Safety Authority, Wageningen, The Netherlands
O-29 **Development, validation and implementation of a GC-EI-Orbitrap HRMS method for routine analysis of pesticide residues in fruits and vegetables**

4:05-4:25 pm **Paul Yang**, MOECC, Etobicoke, Canada
O-30 **Correlation of estrogenicity in wastewater using effect-directed analyses and liquid chromatography-tandem mass spectrometry**

4:30-4:50 pm **Peter L. Morton**, National High Magnetic Field Laboratory/FSU, Tallahassee, FL, USA
O-31 **Contamination of Turmeric with Lead Paint**

4:55-5:10 pm **Poster Awards and Closing** **Orchid Ballroom**

Thursday, July 26, 2018

User Meetings

7:30-9:30 am	**SCIEX User Meeting**	**Orchid 2**
7:30-9:30 am	**Thermo Fisher Scientific**	**Orchid 4**
10:30 am-12:30 pm	**Agilent Technologies User Meeting**	**Orchid 3**

Oral and Poster Presenters

(alphabetical order Last Name, First Name)

Abafe	Ovokeroye	**O-14**
Abel	Ben	**P-85**
Abubeker	Sitra	**O-28**
Antonious	George F.	**P-36**
Armbrust	Kevin	**P-82**
Atkins	Patricia L	**P-33**
Banaszewski	Katarzyna	**P-63**
Belao	Nebebech	**P-41**
Bhardwaj	Laxmikant	**P-64**
Biggerstaff	Daniel	**P-26**
Brahm	Prakash	**P-30**
Broadaway	Bryanna	**P-58**
Cannonier	Shareena	**P-4**
Carlos	Katherine	**P-35**
Carlson	Jules	**P-57**
Chamkasem	Narong	**P-13, P-14**
Chow	Willis	**P-38, P-39**
Christison	Terri	**P-45, P-48**
Cleland	Gareth	**P-92**
Crawford	Lauren M.	**P-8**
Cudjoe	Erasmus	**P-50**
Dahl	Jeff	**P-29**
Dalmia	Avinash	**O-10, P-51**
Delatour	Thierry	**O-12**
Dolata	Lori	**P-46**
Dolata	Lori	**P-47**
Dowgiallo	Anne-Marie	**P-28**
Dusek	Martin	**P-20, P-59**
Ebitson	Michael	**P-90**
Eckert	Matthew	**P-87**
Edwards	Jim	**P-76**
Engel	Marc E.	**P-40**
Faden	Geoffrey	**O-17, P-25**
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ORAL PRESENTATION ABSTRACTS

A-1 Matrix Compatible Coatings in SPME: An Enabling Technology Facilitating Full Automation and On-site Residue Determinations of Food Products

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The availability of state-of-the-art analytical instrumentation offering higher sensitivity and specificity has contributed to an increased range of applications covered by the SPME technique. The presentation will summarize the most underlying aspects in SPME development addressing some of the challenges encountered in the analysis of food samples, with particular emphasis placed on complex sample analysis and rapid screening. In addition, the development of new morphologies of extracting materials and novel sampling configurations as well as approaches compatible with high throughput lab and/or on-site determinations will be outlined. The recent development of matrix compatible SPME coating lead to interesting features experienced during extraction, some of them not anticipated. They are not limited to elimination of fouling and saturation effects during direct SPME of complex samples, but also balance coverage property, enabling "via free form" clean extraction of small molecules widely varying in physical properties leading to some interesting applications. For example, on-site sampling, in-vivo metabolomics, and rapid screening via direct coupling of sample preparation to mass spectrometry were facilitated by this development. Multi-residue method for quantitative analysis of 76 veterinary drugs representing different classes and varying in physical and chemical properties in chicken muscle using SPME/UHPLC-ESI-MS/MS will also be described. Validation demonstrated that the developed method is suitable for fast and reliable quantitative determinations at half or below the maximum regulatory levels. To serve as a guide to potential opportunities for continued innovation in SPME food applications, special emphasis will be placed on the evolution of on-site and in vivo SPME techniques and their feasibility for both targeted determination of organic pollutants and biologically active compounds, as well as for global metabolite analysis.

O-1 Comprehensive study of different approaches for the determination of 15 highly polar pesticides in food matrices. Influence of matrix type on the identification

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Highly polar herbicides such as glyphosate, are well known because of their high effectiveness and low costs. Glyphosate, and many of the other target pesticides and plant growth regulators included in this evaluation are some of the most frequently used pesticides worldwide.

The analysis of highly polar pesticides by a single LC-MS/MS method is very challenging as a consequence of diverse detection and separation behaviour, due to their physical-chemical properties. Nowadays, many companies and laboratories around the world are focusing on the development of new products and analytical methods which make the analysis of these compounds possible in a reliable and robust way. However, although a wide range of methodologies have been developed trying to make the analysis of polar compounds successful, retention time shifts, high matrix effects and rapid degradation of the used columns still limit their utility and their cost-effectiveness.

The aim of this study is the evaluation of a methanol-based extraction method with a clean-up step. For the clean-up step, several sorbents from different brands (EMRd, CAPTIVA, Oasis HLB, Oasis WCX and MCX), with diverse mechanisms of action, have been tested. Three different UHPLC columns have been assessed, in order to determine which one provides the best results in terms of chromatographic separation, peak shape and sensitivity. Besides these tests, different dilution factors for the final extract were applied in order to check the impact on the matrix effect. Matrices from different commodity groups were validated with the optimal method.

O-2 Improved Extraction and Cleanup Prior to LC-MS/MS Determination of Bound and Unbound Dicamba and Dicamba Metabolite Residues in Agricultural Samples

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Dicamba herbicide is used for agricultural weed control in production of many types of crops. To help insure public health and safety, reliable analytical methods are necessary to determine residues of this herbicide and its metabolites in agricultural samples. Because this compound has significant volatility, there is also the need for residue analysis to monitor exposure of crops from drift of applied dicamba. The unbound (free) dicamba can be extracted from agricultural samples in the same manner as most other pesticides using standard QuEChERS methods but these methods may not be suitable for the more polar Dicamba metabolites. Also, dicamba can exist in bound forms (esters or conjugates); determination of the total of bound and unbound residues requires hydrolysis prior to QuEChERS or other manner of extraction. For cleanup, the PSA based dSPE cleanup often employed for QuEChERS analysis is not appropriate for acidic compounds like dicamba and its metabolites because they are strongly retained on the PSA sorbent. A modified QuEChERS extraction protocol, including a hydrolysis step, was therefore evaluated for LC-MS/MS determination of dicamba and dicamba metabolites. Cleanup options (dispersive and pass-through) for the modified QuEChERS method will be discussed.

O-3 Single tube extraction and clean up using solubilized QuEChERS extractants – a miniaturized approach

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We propose a novel enhancement of conventional QuEChERS preparative methodology for the extraction of small amounts of sample using a solution of buffering agents in water to enable pH adjustment and phase separation with organic solvents. To assess the effectiveness of this strategy, organic wheat grain was spiked with a broad-spectrum systemic fungicide. Briefly, 50mg of finely blended grain samples were transferred to 2mL micro-centrifuge tubes and spiked at various concentrations. Acidified acetonitrile was initially added to re-suspend the sample followed by the addition of a near-saturation solution of buffering salts in water. Of significance, a clean-up step was immediately performed in the same tube by adding a mixture of dispersive SPE sorbent in acidified acetonitrile. The mixture was shaken thoroughly and centrifuged using a mini benchtop centrifuge to simultaneously separate undesirable instrumental interferences from the organic solvent. The resulting supernatant was analyzed by gas chromatography-tandem mass spectrometry. Linearity was observed between 1 to 500ng/mL of spiked analytes and correlation coefficients were between 0.990-0.999 for spiked deuterated internal standards and the analyte of interest. The method is characterized by good reproducibility and high recoveries and the cost of sample preparation is considerably decreased due to assay miniaturization. Fast sample preparation time is achieved (< 15mins).

O-4 Surface Enhanced Raman Spectroscopy for Food Colorant Analysis

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We have developed a rapid method for detecting food colorants based on surface-enhanced Raman spectroscopy (SERS) and tested its ability to identify a wide variety of approved and banned artificial and natural food coloring agents currently used in the United States. A simple silver nanoparticle substrate was used to enhance the signal, requiring less than 10 minutes of sample preparation prior to analysis. All of the colorants had distinct SERS signals and could be easily differentiated based on their spectral features and using principal component analysis. Further tests confirmed that SERS is capable of quantifying adulteration with chemically and visually similar colorants, and can detect signals from some artificial colorants down to at least 1 ppm concentrations. Additionally, both artificial and natural colorants could be identified in commercially available food products using the technique. This study establishes a database (N=16) of

commonly used artificial and natural food colorants. The simplicity of the SERS method and its strong effectiveness for detecting colorants indicate that it has great potential to be used for practical applications in this area.

O-5 Food Sensors with Printed, Floating-Gate Transistors

Scott P. White, Ph.D.

Prof. Kevin D. Dorfman and Prof. C. Daniel Frisbie

University of Minnesota

The safety of food products is paramount and ensured through routine analytical sampling for potential contaminants. Next-generation sensors are designed to gather this information with less resources such as time, labor, or cost. In this work, we applied Floating-Gate Transistors (FGTs) to food sensing by synergistically combining expertise in printed electronics and microfluidics. The FGTs rapidly transduce the binding of biomolecules to an aptamer or antibody functionalized electrode. The resulting potentiometric perturbation depends on the intrinsic properties of the analyte, bypassing the need for labeling reagents. Additionally, the incorporation of microfluidics increases analyte binding rates without the need for sample pre-concentration. The resulting analysis time is ~30% lower than conventional techniques such as ELISA when applied to DNA, proteins, and/or small molecules. The scalability and usability of the FGT platform also enables streamlined production and throughput which is the focus of current and future development. This talk will focus on the application of FGTs to gluten and glyphosate detection from real samples and its ability to multiplex numerous FGT pixels into an array for simultaneous detection and sourcing of complex contaminants such as gluten.

O-6 Plasma Assisted Reaction Chemical Ionization: A Sensitive Ion Source for Elemental Detection and Quantification of Residues

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Elemental mass spectrometry (MS) coupled to chromatography offers facile screening and quantification workflows. For example, element-targeted screening rapidly identifies retention times of compounds having known or suspected elements in complex mixtures. More importantly, robust elemental ionization provides elemental concentrations of the compounds with minimal matrix effects, offering quantification without compound-specific standards. These characteristics have been utilized extensively for speciation of metals using inductively coupled plasma (ICP)-MS. However, extending this powerful analytical workflow to organic compounds has been hindered by the low sensitivity of ICP-MS for non-metal analysis. In particular, many residues include difficult-to-detect elements such as halogens. For example, over 30% of drugs contain F or Cl. More than 50% of pesticides are halogenated while fluorinated and organophosphorus contaminants have been increasing in food and environmental samples. We will present plasma assisted reaction chemical ionization (PARCI) as a new approach to address the shortcomings of elemental quantification for non-metals. In PARCI, analytes separated from a chromatographic column are directed into a plasma where neutral precursors are generated. Chemical ionization of these element-specific neutrals in the plasma afterglow provides sensitive detection of the elements. Importantly, PARCI is implemented using molecular mass spectrometers, alleviating the need to have a dedicated elemental MS. This strategy further facilitates integration of elemental and molecular MS for comprehensive analysis of complex mixtures. We will present examples for detection and quantification of GC and LC separated organohalogens.

O-7 Pesticide analysis in complex matrices by new GC and LC MS/MS improved platforms

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In spite of the high number of published works on pesticide Multi residue Methods (MRMs) based on GC and LC-MS, the analysis of complex matrices remains a great challenge in food control laboratories. Complex matrices can be defined

as those matrices that provide intense ion suppression or enhancement with an appreciable number of analytes in comparison with the responses obtained with pure solvent.

Various approaches can be applied to overcome the negative impact of the high number or concentration of the co-eluted molecules. These methods can be based on the use of improved sample preparation methods or the use of more efficient mass spectrometry platforms.

The application of new sample preparation methods -which can be very different than the well-standardized ones- creates difficulties in routine laboratories as they increase the workflow of analysis. These methods have to be validated and, in many cases, additional complications can appear. The use of more sensitive mass spectrometry platforms represents a good balanced solution to improve the difficulties commented without an extensive validation work. High-resolution accurate-mass can be additionally an acceptable approach considering the increase in selectivity obtained by these techniques.

The present work is focused on the analysis of pesticide residues in spices by GC and LC-MS. Various analyzers such as; Triple quadrupole, time of flight and orbitrap are evaluated for their application for MRM. Additionally the proposed methods have been applied to real samples to facilitate a more comprehensive evaluation of their efficiency.

O-8 Utilizing GCxGC-TOFMS for Quantitation of Cannabis Related Pesticides in Hops QuEChERS Extract.

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Matrix deleteriously affecting quantitation accuracy of pesticides in food commodities has been well established. As a result utilizing matrix matched standards has become the industry accepted practice and establishing a matrix blank is relatively straight forward; simply screen an extract of that commodity for the targeted compounds. Establishing a matrix blank for cannabis is considerably more challenging due cost, varietal differences, innate complexity of cannabis as matrix and regulatory limitation on transporting raw product or extracts. For these reasons, some labs have opted to use standard curves in solvent for quantitation of pesticides. Given the regulatory environment is unlikely to change in the foreseeable future, establishing inexpensive, legally transportable matrix blanks would seem to be a priority to ensure accurate pesticide detection/quantitation and to bring cannabis testing in-line with other common commodities. In this presentation, we will evaluate hops as a possible stand-in for cannabis as a matrix standard. Hops are inexpensive, wildly available, freely transportable across the United States and like cannabis extracts, hops extracts are extremely complex. To combat this matrix load, GCxGC was implemented to chromatographically resolve the target pesticides from the matrix interferences. Evaluations of solvent verse matrix calibration curves as well as detection limits for commonly used pesticides will be shown as well as the advantages provided by GCxGC separations.

O-9 Comprehensive Determination of Unregulated Pesticides in Cannabis Flower using Tandem Mass Spectrometry

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No legal tolerances for pesticide residues on cannabis have been established in the United States, with each state setting independent regulations for the pesticide residue levels allowed in cannabis products. Each state has a different list of regulated pesticides which are required testing for cannabis, but no broad pesticide testing methods are being performed to detect illegal application of pesticides on cannabis crops. A method has been developed using LC-MS/MS and GC-MS/MS to screen for over 350 different agricultural pesticides in cannabis flower tissue. This method was used to screen over 100 different cannabis samples from growers across the state of Oregon, to demonstrate the scope of off-label pesticides which are currently being used on cannabis crops.

O-10 Analysis of all pesticide residues in cannabis regulated by California and Oregon state using LC/MS/MS with Electrospray and APCI source

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Over 30 states in the U.S. have legalized the use of recreational or medical cannabis because of therapeutic benefits for ailments such as cancer, multiple sclerosis, and ALS. Since chronic exposure to pesticides in cannabis pose serious health risks, pesticide analysis in cannabis is important. Apart from Oregon state regulatory limits for about 65 pesticides in cannabis, the state of California has issued more stringent regulatory limits for about 72 pesticides residues in cannabis flower and edibles. Normally pesticide analysis in cannabis and other food matrices is done by both GCMS and LCMS since some nonpolar and chlorinated pesticides are difficult to ionize with electrospray and APCI ion sources used in LCMS systems. Two different LCMSMS methods with electrospray and APCI source were used for low level analysis of all pesticides (including the very hydrophobic and chlorinated pesticides analyzed by GCMS) in cannabis. The overall sensitivity for most of the pesticides, including those that are normally analyzed by GCMS was between 1-300 ppb in cannabis, well below regulatory limits set by the state of Oregon and California. The ability to screen and quantitate all 72 pesticides, including the very hydrophobic and chlorinated GCMS amenable, in cannabis with LCMSMS only with dual ESI/APCI source makes this a novel way of screening and quantitation of pesticides in cannabis and different matrices with a single instrument. Long term stability data for pesticide analysis in cannabis showed that response RSD over 1 week for majority of pesticides was between 2 to 10 %.

O-11 Cannabis Sativa pesticides, aflatoxins, and potency by LC/MS/MS: one extraction, one analysis

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Presently eight USA states and the District of Columbia have legalized recreational use of marijuana. An additional 20 states have legalized marijuana for medical use. With these changes in law the need to regulate safe use and authenticate quality of the substance has evolved to include testing for pesticides, mold (aspergillus) and its secondary metabolites, mycotoxins. In addition, potency testing in states that allow its medical use is a requirement. The ability to combine each of these analyses into one methodology that is accurate, precise and robust would be advantageous. However, major portions of the plant (especially buds), extracts, concentrates and edibles constitute a very complex matrix that presents interferences for the trace analysis of pesticides and mycotoxins—even for MS/MS.

Samples of the bud are collected and extracted using a QuECHERs procedure modified to include additional cleanup with a flow-through cartridge. The extract is analyzed using LC-MS/MS, including all LC-MS amenable pesticides on the California list, the five regulated mycotoxins: ochratoxin A, aflatoxins B1, B2, G1 and G2, and the major cannabinoids (THC, THCA, CBD, CBDA, CBG, and CBN) in the same run. Since the cannabinoids are found in the percent levels, ^{13}C isotopes of these are used to develop MRM transitions that will not saturate the detector while analyzing trace pesticides and mycotoxins at ppb levels.

O-12 Matrix effect-monitored screening of a hundred veterinary drug residues in food by liquid chromatography-triple quadrupole mass spectrometry

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The presence of veterinary drug residues in food products constitutes a potential health risk for consumers as they might induce various effects such as allergic reactions, carcinogenic or teratogenic mechanisms or induce antimicrobial resistance. In particular, antimicrobial resistance is considered as an increasing threat to global public that now requires adequate actions across governments and society. Codex Alimentarius, European Union and several countries have established maximum residue limits for veterinary drugs in food with the intention to limit consumer exposure to such residues. We have developed of liquid chromatography-tandem mass spectrometry (LC-MS/MS) method for the analysis of a hundred veterinary drug residues in a broad range of food commodities, including raw materials and semi-processed

ingredients. Due to the low number of non-compliant samples that has been evidenced by recent surveys in the USA and the Europe (< 1%), we have chosen a screening approach for an effective clearing of compliant commodities. Our screening approach will be shown and compared to other strategies available from the literature. Ultimately, the benefit of the approach in terms of low rates of false negatives will be highlighted.

O-13 Moving from targeted towards non-targeted approaches

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Truly non-targeted approaches focusing on the generic detection of exogenous compounds in complex matrices like food or environmental samples seem to remain a rather distant goal for residue analysts. Yet, detecting compounds derived from targeted analytes is a more realistic strategy. This may include the monitoring of degradation products of pesticides or metabolites of veterinary drugs. Such compounds are frequently not available as commercial reference substances. Hence, the HRMS based detection of a drug and the corresponding metabolites (e.g. glucuronidated or sulphonated) in tissue sample is a solid proof that the sample has not been accidentally contaminated, but that the drug has truly been ingested by the animal. Such semi-targeted techniques may also be extended to focus on the detection of all compounds containing specific atoms (e.g. halogens, sulphur etc.).

The road leading to non-targeted approaches is likely to be based on data independent acquisition modes. Such techniques aim at providing comprehensive MS/MS or ion-mobility/MS "landscapes". The first dimension (precursor ion) may be "sliced" into a number of adjacent mass ranges (SWATH) or it may be produced by a continuous scanning device.

Data mining a two-dimensional space requires powerful software tools. This certainly must include the conventional data processing tools for quantitative residue analysis, but in addition should contain algorithms permitting deeper data mining capabilities (e.g. spectra deconvolution and in-silico fragmentation). The enormous volume of produced data has not only to be acquired, but also stored and processed. This still presents a bottleneck for current IT technologies.

O-14 Development and validation of a confirmatory method for the determination of stilbene estrogens in Ostrich serum

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A simple and accurate ultra-high performance liquid chromatography tandem mass spectrometry (UHPLC-MS/MS) method was developed and validated for the first time as a confirmatory method for the simultaneous determination of stilbenes – hexestrol and diethylstibestrol in serum. Extraction was based on a simple acid denaturation of protein followed by liquid-liquid extraction using methyl tert butyl ether (MTBE). Extracts were directly injected into the UHPLC-MS/MS without further purification. Excellent recoveries in the range 82 – 99 % and 91 – 128 % were obtained for hexestrol and diethylstibestrol, respectively. Both within-day repeatability and between-day reproducibility were generally satisfactory with RSD < 20 %. The linearity of the internal standard based matrix-matched calibration curve measured as the coefficient of regression (r^2) was generally > 0.99 for both hexestrol and diethylstibestrol. Both matrix effect and uncertainties associated with sample preparation and instrumental analysis were significantly reduced with the use of a deuterated compound (hexestrol-d4) as internal standard. The limits of detection (LOD) and limit of quantitation (LOQ) were 0.09, 0.08 ng/ml and 0.28, 0.25 ng/ml respectively for hexestrol and diethylstibestrol. The method was found to be suitable for the simultaneous determination of hexestrol and diethylstibestrol in serum.

O-15 Accurate Quantitation and Analysis of Nitrofurans and Phenicols in Seafood by UHPLC-MS/MS: Proficiency Testing and State Regulatory Samples

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Previously we introduced a new method for screening four nitrofuran metabolites: 3-amino-2-oxazolidinone (AOZ),

3-amino-5-morpholinomethyl-2-oxazolidinone (AMOZ), semicarbazide (SCA), and 1-aminohydantoin (AHD), and well as phenicols: chloramphenicol and florfenicol in seafood for tilapia and shrimp. Liquid-liquid extraction and ultra high-performance liquid chromatography-tandem mass spectrometry (UHPLC-MS/MS) were used to analyze the samples and quantitated with in solvent, isotopically labelled standards. The limit of detection (LOD) was established at 1 ng/g for all nitrofurans, and 0.25 ng/g for the phenicols. Since then the method has passed proficiency tests for nitrofurans and phenicols and currently analyzing various seafood commodities such as shrimp, tilapia, swai, crab, and frog legs. Our results show that majority of the banned veterinary drugs have been found in frog legs in over 90% of the frog legs samples analyzed.

O-16 HPLC Method for Quantification of Mycotoxins in Animal Matrices, Food and Feed

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Mycotoxins are naturally occurring, secondary metabolites of molds and fungi. They are common contaminants of food and feed commodities and have various adverse effects on human and animal health. Contaminated feeds often contain multiple different mycotoxins. Formation of mycotoxins depends on the type of matrix as well as growing, harvesting and storage conditions. The present method is capable of analyzing Deoxynivalenol, Aflatoxins, Fumonisins, Ochratoxin A, and Zearalenone in a single run using HPLC with post-column and photochemical derivatization, using fluorescence and UV detection. This method and instrumentation allow for interference-free detection of Aflatoxins at the low ppb level, so we additionally developed a fast HPLC method to sensitively and robustly analyze Aflatoxins in peanut butter, ground peanuts and in herbs and spices. Another mycotoxins group, Fumonisins, are suspected human carcinogens and are toxic to pigs, poultry and horses. Fumonisins are produced by Fusarium moniliforme that grow on corn and other commodities. Many countries set limits on the presence of Fumonisins in foods and feed; testing of raw crops and finished products is regularly performed. The US FDA sets total Fumonisins limits in human foods between 2-4 ug/g and in animal feed between 5-100 ug/g. As Fumonisins do not have a chromophore and do not fluoresce, derivatization is needed to achieve the required sensitivity of detection. We developed a fast and sensitive HPLC method with post-column derivatization to meet this need, capable of analyzing Fumonisins in grains and animal feed at levels as low as 0.01 ug/g using a shorter run time than the full multi-residue mycotoxins method.

O-17 Fast, Highly Sensitive Analysis of Mycotoxins Using Superficially Porous Particles

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People trust that the food they are eating is safe and free from harmful contaminants. However, if proper storage conditions are not maintained, the formation of molds and fungi is possible. In turn, the fungi can produce toxic secondary metabolites called mycotoxins, which can threaten human and animal health alike. Hundreds of mycotoxins have been identified and the levels of some of these are regulated across the globe. An example of a regulated mycotoxin is patulin, which is formed by several different molds that can be found especially on apples, but also on other fruits, such as pears, grapes, and peaches. The presence of patulin can also be found in juices made from these fruits.

Since patulin does not fluoresce, another sensitive detection method must be used. The combination of HPLC with either UV or MS detection has been optimized using columns of superficially porous particles (SPPs) with the goal of high throughput. SPP columns are known for their robust performance and their ability to run at elevated flow rates while maintaining resolution making them well suited for laboratories demanding both speed and resolution. This presentation will highlight the advantages of using SPP columns for mycotoxin analysis using a variety of fruit and fruit juice matrices.

O-18 What are we missing? Metabolomics, toxin analysis, and new compound discovery from marine and freshwater HAB species

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The frequency and severity of aquatic harmful algal blooms (HABs) are increasing with alarming results. These blooms produce a variety of potent toxins such as the microcystins that comprise a group of over 200 cyclic peptides with many unique chemical features and a range of biological activities. However, only 4 of these are routinely monitored by regulatory agencies. In marine HABs, a range of toxins with exquisite levels of potency are often present leading to severe episodes of intoxication in people and wildlife. In addition to toxins, these same organisms produce other compounds with potentially therapeutic bioactivities. UPLC-HR-ToF-MS based untargeted metabolomics analysis and molecular networking combined with traditional chromatographic isolation and structure elucidation is a powerful approach to understand the chemical production and correlated biological activity of both known toxins and other biologically active compounds produced by these CHAB species.

In this study, we analyzed the organic extracts from laboratory cultures of HAB-forming freshwater cyanobacteria and marine dinoflagellates using a metabolomics approach. From this analysis and subsequent isolation and structure determination, we have discovered many new compounds with diverse chemical structures and a variety of biological activities. Select laboratory strains have also now been cultured to create a parallel library of these high-value toxins completely labeled with ^{15}N isotope. These surrogate standards enable increased monitoring sensitivity and accurate structure assignment through fragmentation analysis. Results of the analysis, structures, ^{15}N labeling, and biological activities will be presented.

O-19 Analytical Methods and Reference Materials for Marine Toxins

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Marine toxins, such as those produced by harmful algae, have negative impacts on human health, commercially associated industries, and on recreational activities around the world. The wide range and complexity of toxin analog profiles means that measurement and regulation is a significant challenge. Sound measurements rely on good analytical methods which are supported by high-quality standards and reference materials (RMs). This presentation will give an overview of work carried out at the National Research Council Canada (NRCC) to develop analytical methods and RMs for a range of marine toxins including the major lipophilic (okadaic acid and dinophysistoxins, azaspiracids, yessotoxins, pectenotoxins, cyclic imines) and hydrophilic (paralytic shellfish toxins, domoic acid, tetrodotoxin) toxin groups. General concepts of traceability will be introduced along with the approaches taken to produce certified RMs for marine toxins. These RMs have been valuable in development and validation of a range of analytical methods for various toxin classes. Examples provided will include targeted low-resolution and non-targeted high-resolution LC-MS methods for general classes of lipophilic and hydrophilic toxins, direct and rapid methods of analysis for domoic acid, and finally recent work on development of RMs and methods for measurement of tetrodotoxin as an emerging issue in bivalve shellfish.

O-20 Insecticide Exposures and Human Health

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Modern insecticides – those synthetically derived since WWII -- are a unique class of chemicals specifically designed to be lethal which we purposely apply to lawns, gardens, crops and residential areas. Pesticides also have become a critical public health tool helping to stem outbreaks of vector-borne disease such as dengue and malaria and providing for a plentiful food supply. In general, insecticides act by altering the neuronal signaling pathways at the terminus or axonal membranes, and since insects and humans share these signaling pathways, the potential exists for pesticides to affect humans similarly. Newer data suggest that in addition to the acute toxicities of pesticides in humans, that alternate dopaminergic and serotonergic pathways may be involved in more subtle neurological alterations that result in neurocognitive deficits including more pervasive ADHD, lower cognitive and motor functioning and decreases in IQs. The pre- and peri-natal period of life seems to be the most critical windows of exposure. In our Thai farmworker birth cohort, we collected time-resolved pesticide exposure data during pregnancy and evaluated neurodevelopment at birth and up to 3 years of age. We found that 1st trimester exposures to pesticides were linked to autonomic activities such as number of abnormal reflexes. Emotion, attention and motor skill deficits were linked to 2nd trimester exposures. Our data add to the growing body of evidence implicating pesticide exposure in neurodevelopmental effects.

O-21 Analysis of Cyanotoxin and Algal Toxins: Data gaps, Challenges, and Advancements

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Cyanotoxins and algal toxins are naturally occurring compounds produced by a range of photosynthetic cyanobacteria and algae. These compounds are of concern due to toxic cyanobacterial and algal blooms' association with poisoning events of animals and humans. Additionally, there is a perception that the blooms are increasing in size and frequency in surface waters globally. Analytical measurement of the toxins has been historically challenging because of a lack of certified reference materials, the number of toxins, analytical limitations, variations in toxin chemistry and matrix, representative sample collection, and study design. Methods currently used range from bioactivity assays to high resolution mass spectrometry. Analysis of exposure in tissues, sediments and soils represent further complication to achieving suitable quantitation for toxin measurement. Data gaps, challenges, and advancements in analytical chemistry will be discussed.

O-22 One Health Approach to Chemical Residues in Our Environment

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One Health is a concept which acknowledges that the health of people, animals and the environment are closely connected. The goal of One Health is to expand interdisciplinary collaborations worldwide to promote the health of humans, animals, and the environment. During this session, three speakers will present scientific studies that meet this standard. One Health applications include monitoring natural or synthetic chemical residues in people, animals, and the environment to accurately assess acute and long term impacts in these complex biologic systems. As the human population continues to grow while essential resources remain finite, the need for high quality chemical residue studies continues and becomes more pressing. Understanding what chemicals we are eating, drinking, and breathing in, and how they interact with our bodies is imperative. The introductory presentation will provide a brief history of One Health. In addition, recent examples of One Health studies involving wildlife, domestic animals, plants, and the environment, and a brief description of the applications of these studies will be used to highlight how interconnected the health of people, animals, and the environment are.

O-23 Integrating GC-TOF exposome profiling and genetic disease screening to provide a holistic perspective on honey bee health

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Global honey bee (Apis mellifera) populations have been declining for more than a decade, and very few studies integrate multidimensional data to assess bee health. Integrating genetic risk, chemical and pathogenic exposure, nutrition, and individual's internal biochemistry (the metabolome) presents an enormous challenge. To study the genome (the expression of ones' genetic code), sophisticated and harmonized tools and bioinformatics have been developed to address these complex biological questions using big data (omics) approaches. More recently, exposomics methodologies have been developed to characterize the environment's contribution to health risks. The exposome considers how an organisms internal chemical environment responds to external exposures, and is the compliment to the genome. Exposomic characterization relies on discovery-based (non-targeted, hypothesis agnostic) methodologies to generate new hypotheses, followed by independent targeted analyses to validate the findings and identify associations of exposure events with biological response pathways. In this study, we integrate exposome profiles generated by gas chromatography time-of-flight mass spectrometry (GC-TOF) with semi-quantitative PCR disease screening to establish chemical signatures and associations between healthy and unhealthy honey bees. This presentation will detail the data-collection and data-analysis methods used to characterize the exposome of the western honey bee, and illustrate a putative mechanism that may play a significant role in pollinator decline.

O-24 Reliable and Cost-Effective Analysis of Bisphenol A and Nonylphenols in Foods and Beverages

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Bisphenol A (BPA) and nonylphenols (nonylphenol isomers, NPs) are environmental contaminants and prominent examples of endocrine disrupting chemicals that adversely impact the human reproduction and neurobehavioral development. The contamination of food products by BPA and NPs raise public and regulatory concerns. We established a simple, reliable, and cost-effective approach to quantify BPA and NPs (individually or together) in various food and beverage matrices. The methodology combines a salting-out assisted liquid/liquid extraction with acidified acetonitrile and the freeze-out removal of the co-extracted lipids before the analysis by LC-MS/MS in negative ESI mode. It has been validated on various food matrices, including commercially packaged beverages, infant formula products and related ingredients, with acceptable accuracy and precision. The limits of quantification (LOQs) are 0.3 ng/g (BPA) and 10 ng/g (353-NP). Satisfactory recoveries were obtained for both BPA and 353-NP in the range of 92 - 127% in the validated food matrices at several fortification levels. The samples spiked at the LOQ level over three days in each food matrix yielded an interday precision in the range of 1.9 - 11.5%. Moreover, the performance of this method met the AOAC standard method performance requirements (SMPR) 2017.018 for "Determination of Bisphenol A (BPA) in Commercially Packaged Ready to Consume Carbonated and Noncarbonated Water and Non-Alcoholic Beverages", and the method was approved AOAC Official First Action method for BPA analysis in December 2017.

O-25 Analysis of organophosphate esters in meats and fish

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Organophosphate esters (OPEs) are high volume production chemicals widely used as plasticizers and anti-foaming agents, and most recently as flame retardants in textile, furniture, electronics, building, automobile and other industries. OPEs easy liberate from treated materials, and hence are ubiquitous in the environment. While there is not enough knowledge on OPEs' toxicity, some, like tris(2-chloroethyl) phosphate, are classified as carcinogenic and banned by the European Union. Published studies so far have reported OPEs in air, water, soil, sediments, biota, and humans, but no data on OPE levels in foods consumed in the USA were found. In this study, we have developed a method for analysis of the 14 most widely used OPEs in chicken, swine, cattle, salmon and catfish muscle. The method was based on QuEChERS extraction with acetonitrile and simultaneous salting-out. An aliquot of the extract was filtered and analyzed by UHPLC-MS/MS for 14 OPEs and two isotopically labeled internal standards, while another aliquot was subjected to automated solid phase extraction (SPE) mini-columns cleanup with Instrument Top Sample Preparation (ITSP) and analyzed with low pressure (LP)GC-MS/MS for 9 GC-amenable OPEs. The developed method was validated in the five selected matrices at four spiking levels (5, 10, 20 and 40 ng/g), and satisfactory recoveries (70-120%) and RSDs (<20%) were achieved for 13 analytes. OPEs' ubiquitous presence in laboratory environment and materials presented a challenge to their accurate quantitation. The analysis of meat and fish samples from US markets is underway to generate data on their presence in these foods.

O-26 Untargeted Screening and Prioritization of Halogenated Chemicals in the Environment

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The vast majority of toxic chemicals in the environment remain unidentified. Untargeted screening and prioritization of environmental chemicals is critical to more completely understand their potential health risks. A series of chemistry and biology tools were developed in my group to screen and prioritize environmental chemicals (i.e. halogenated compounds) in an unbiased fashion. Complementary High-Resolution Mass Spectrometry (HRMS) based methods were developed to enhance the coverage for detection of various halogenated compounds: Data-Independent Precursor Isolation and Characteristic Fragment (DIPIC-Frag) method, was developed for selective identification of iodinated and brominated compounds in the environment; Putative lock mass re-calibration algorithm and Bayesian algorithm

were combined for robust detection of chlorinated compounds; Mass defect filtering algorithm in high-resolution MS^1 and MS^2 spectra was used to screen fluorinated compounds. The developed methods were applied to different environmental matrices including sediments, drinking waters and house dusts. Thousands of known and unknown halogenated compounds were detected. Specifically, brominated azo dyes were identified as the most abundant compounds in house dust. Further toxicology studies on zebrafish revealed compound-specific toxicities of brominated azo dyes, at both phenotype- and proteome-level. These studies indicates that HMRS based methods could be applied to screen and prioritize unknown compounds in the environment, in an unbiased fashion.

O-27 Non-targeted Analysis of Emerging Environmental Contaminants by Data Independent Acquisition LC-MS/MS

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Emerging environmental contaminants such as pharmaceutical and veterinary drugs result from wastewater treatment plant effluent as well as runoff from agriculture land fertilized with animal manure. These compounds are of great concern to regulators due to their potential impact on human health and the environment. Advances in high-resolution Orbitrap and Q-TOF mass spectrometers (MS) has allowed for non-targeted screening methods to be developed. We have developed data independent-acquisition (DIA) methods on a Q-Exactive Orbitrap LC-MS/MS system for both positive and negative electrospray ionization. To validate these methods, we have applied a spectral counting approach to calculate the selectivities of DIA as compared to other LC−MS acquisition modes such as HRMS alone or in combination with all-ion-fragmentation (AIF) or data-dependent-acquisition (DDA). Our DIA method was then used to analyze hundreds of surface water samples collected from sites in Ontario, Canada. Each sample was screened for the presence of 97 pharmaceuticals and 171 pesticides on our current target list. We reliably detected 34 pharmaceuticals, 42 pesticides and some artificial sweeteners from the surface water samples. The non-targeted data was also digitally archived for retrospective analysis. The ability to quickly assess this data has been valuable for risk assessments of new contaminants of concern without re-running samples. For example, the presence of the chlorpyrifos metabolite TCPy was assessed in minutes without it being on the target list. This compound would have been missed by traditional targeted analysis and demonstrates the power of non-targeted analysis and archiving data for retrospective analysis.

O-28 USDA's Pesticide Data Program – Who Uses the Data

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USDA's Pesticide Data Program(PDP) is a national pesticide residue monitoring program. In collaboration with State Department of Agriculture, PDP manages the sampling, testing and reporting of pesticide residues on agricultural commodities in the U.S. PDP's sample collection is statistically-based with laboratories using current testing methods and instrumentation. PDP data are primarily used by the U.S. Environmental Protection Agency for dietary risk assessment to make informed pesticide reregistration decisions. Data users also include the U.S. Food and Drug Administration for tolerance violation surveillance, crop protection companies for refining pesticide risk assessment and in the CODEX work for benchmarking Maximum Residue Levels (MRL's). The USDA's Foreign Agricultural Service also uses PDP data to resolve pesticide residue violation in international trade.

O-29 Development, validation and implementation of a GC-EI-Orbitrap HRMS method for routine analysis of pesticide residues in fruits and vegetables

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Fast and comprehensive full-scan high resolution accurate mass (HRMS) analysis has recently become available as an alternative for the governmental and private pesticide residue laboratories responsible for providing residue data for enforcement, the EU-coordinated monitoring program or official EU import control program. Nowadays, high resolution

instruments provide an unique combination of excellent mass-accuracy, high resolving power, as well as good sensitivity and selectivity, allowing laboratories to analyse the most challenging matrices with a practically unlimited scope. Additionally to the high number of target analytes, the technique takes advantage of optional identification of unknowns and retrospective analysis. However, every technique has some limitations.
This poster will discuss the development, validation and routine application of the quantitative method for the analysis of pesticides in fruits and vegetables and more difficult matrices such as tea, spices and herbs by the GC-Orbitrap system. Analysed extracts were prepared using the NL-extraction method. Recovery samples were spiked at three concentration levels (5, 10 and 20 ug/kg) with 310 pesticides and analysed within a 25 minutes chromatographic run.
The EU SANTE AQC document criteria for HRMS identification will be critically evaluated, based on the full validation data. Routine analysis results of the GC-Orbitrap will be compared with GC-MS/MS Triple Quad results, as to sensitivity (method LOD, LOQ, LOI), selectivity and false-positives/negatives, for the analysis of fruits and vegetables. Typical examples of monitoring and survey results will be shown.

O-30 Correlation of estrogenicity in wastewater using effect-directed analyses and liquid chromatography-tandem mass spectrometry

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The mobilization of anthropogenic trace organic contaminants (TrOC) into the environment from run-off after land application of municipal biosolids is a concern for municipalities, regulators and the research community. Field studies focusing on estrogens, a class of endocrine disrupting compounds (EDCs) and subclass of TrOC, have found these and other EDCs in both the run-off, tile drainage from fields that have received biosolids, and in vegetables grown on biosolids amended sites. There is good evidence that effect-based analysis, such as in-vitro single mode activity bioassays similar to the Estrogen Receptor Chemical-Activated LUciferase gene eXpression (ER-CALUX) method can be used to complement chemical based analysis and facilitate workload of liquid chromatography-tandem mass spectrometry (LC-MS/MS) based analysis. In this study, biosolids samples collected from local waste water treatment plants were prepared and aqueous extracts (used to emulate runoff) were analyzed by ER-CALUX at the low part-per trillion level (ng/L, ppt) method detection limits (MDL). These samples were further analyzed using LC-MS/MS for target compounds of known estrogenic effects (e.g., bisphenol A (BPA), equilin, estrone (E1), b-estradiol (b-E2), estriol and 17-a-ethinylestradiol (a-EE2)). Quantitative LC-MS/MS results were acquired with MDLs in the low ppt range; and allowed for a direct correlation to the ER-CALUX results. Due to the minimal quantity of a-EE2 and b-E2 found, it was difficult to establish a direct correlation between ER-CALUX and a-EE2 and b-E2 results. Other target compounds such as BPA and E1 were also found by LC-MS/MS, and were used to correlate the effects observed by the ER-CALUX.

O-31 Contamination of turmeric with lead paint

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Turmeric is a common spice in southern Asia and exported globally. A recent study in Bangladesh showed that 78% of 309 children aged 20-40 months had blood-lead (Pb) levels exceeding the CDC reference level of 5 µg/dL (Gleason et al., 2014). The entire Bay of Bengal suffers intense anthropogenic perturbation, and Pb-contaminated turmeric was identified as the most probable cause of the widespread poisoning, but a plausible mechanism of contamination was never conclusively determined despite anecdotal reports of adulteration with lead chromate ($PbCrO_4$), a yellow paint pigment. We reprocessed and reanalyzed subsamples of the original Gleason et al., 2014 Bangladeshi turmeric samples to (1) independently validate the original Pb concentrations, (2) produce an expanded suite of potentially toxic trace elements (e.g., chromium, Cr), and (3) compare the Pb isotopic composition of the contaminated turmeric with those of anthropogenic and natural sources of Pb to the Indian Ocean. Our independent Pb concentration measurements showed excellent agreement (slope=0.9885, r^2=0.982) with those of the original study. However, while the Cr analysis in the original study proved inconclusive, our reanalysis using HR-ICP-MS revealed a 1:1 molar relationship between Pb and Cr, consistent with adulteration of turmeric by the aforementioned lead chromate pigment. Furthermore, the comparison of Pb isotopic compositions showed that the turmeric Pb is not consistent with Pb in industrial aerosols from the Bay of Bengal. All lines of evidence point towards intentional adulteration of turmeric with lead paint, possibly to improve the color and therefore value of an otherwise inferior turmeric product.

POSTER ABSTRACTS

P-3 Engineering of a Biodegradable Polymer for Treatment of Various Water Systems

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Due to an increased number of polluted water and wastewater systems within the United States, more innovative and feasible solutions must be explored to address contamination due to urban runoff, agricultural waste, and wastewater. This presently proposed work should provide novel science and engineering results and, moreover, a potentially transformative environmentally sustainable water remediation technique that would benefit current society and provide solutions for environmental challenges in the future. US Patent No.: US 8,519,061 B2 was developed for a controlled release chemical oxidation polymer system to remediate wastewater. This biodegradable polymer including potassium permanganate, otherwise known as the "environmental pill", is able to slowly release chemical oxidants at controlled rates and effectively reduce bacteria concentrations in wastewater for extended periods of time. The novelty of this method is not only in the remediation of various water contaminants in the state of Florida; but in the absence of any chemical or contaminant residual or the need for external power or equipment for the process. Through the use of gas chromatography/mass spectrometry (GC/MS) analyses, the expected results of the proposed research will show that the encapsulation of chemical oxidants is expected to treat wastewater, surface water and groundwater systems while degrading potential contaminates for remediation as well as resulting in bacterial inactivation while leaving surrounding environments unharmed. This technology can potentially provide high pay-off results and thus reduce environmental, health and safety risks to communities in various water sources both in the U.S. and throughout the world.

P-4 Assessing the Solid Phase Adsorption Toxin Tracking (SPATT) Bag Method for the Determination of HAB Biotoxin Concentrations in Two Estuarine Ecosystems

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Harmful algal blooms (HABs) are the overgrowths of algae that cause harm by toxin production or by biomass accumulation. HABs are influenced by the availability of sunlight, carbon-dioxide, and nutrients. Other factors include temperature, salinity, wind, water-depth, and grazing predators. Therefore, changes in the Earth's climate will impact the production of HABs. The goal of this study was to determine HAB biotoxin concentrations in Apalachicola National Estuarine Research Reserve (ANERR) and Grand Bay National Estuarine Research Reserve (GNERR) and to determine the correlations to water column nutrient concentrations. Solid Phase Adsorption Tracking (SPATT) bags assessed the amount of dissolved biotoxin levels of domoic acid in seawater. Nutrient and other data will be obtained from the NERR's System-Wide Monitoring Program (SWMP). Dissolved biotoxin extracts were measured using bioassays. A spiking protocol measured the efficacy of the porous resin, where salinity, humic acid, and domoic acid concentrations were tested in deionized water, artificial salt water, and estuarine collected water. Using 1L Erlenmeyer flask, SPATT bags were exposed to the various treatments for 2 weeks and ELISA measured toxin uptake, while spectrophotometry measured humic acid levels. Results indicated that domoic acid concentrations were detected in both estuarine ecosystems. Pseudo-nitzschia pseudodelicatissima was identified as the HAB species. Spiking efficacy test showed that the SPATT bag's resin can collect low levels of domoic acid, while humic acid levels absorbed best at 600nm. This study sheds light on some factors that influence HAB production in estuaries, and contributes to the greater goal of maintaining healthy oceans.

P-5 A rapid single tube extraction and clean-up for the detection of organochlorine pesticides in human milk

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The presence of pesticides in human milk is of great concern due to the potential health effects on breastfed infant. The pesticide levels in human milk can be used as unique markers to reflect pesticide use in environment and to assess human exposure. However, the ability to monitor these pesticides at trace levels is challenging due to limited sample volumes. Here we propose a novel enhancement of the conventional QuEChERS preparative methodology for the extraction of these pesticides from sample volumes ranging from 50-200 µL. To assess the effectiveness of this strategy, a pooled human milk sample was spiked with a mix of organochlorine pesticides (OCPs). Briefly, human milk was aliquoted into 2mL micro-centrifuge tubes and spiked at various concentrations. Acidified acetonitrile was initially added to re-suspend the sample followed by the addition of a near-saturation solution of buffering salts. A clean-up step was immediately performed in the same tube by adding a mixture of dispersive SPE sorbent in acetonitrile. The mixture was shaken thoroughly and centrifuged using a mini benchtop centrifuge. The resulting supernatant was analyzed by gas chromatography-tandem mass spectrometry. Linearity was observed between 1 to 100 ng/mL of spiked analytes and correlation coefficients were between 0.990-0.999 for spiked internal standards in all samples. The 50 µL extracted samples had lower limits of detection, higher peak responses and improved chromatographic peak shapes of OCPs compared to other samples. This shows that trace level residues can be extracted from limited samples using the miniaturized assay whilst maintaining the “matrix-effect”.

P-6 Investigating emerging organic contaminants in harvested rainwater (HRW) via Co-Created Citizen Science

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Today, about 40% of the global population lives in arid and semi-arid environments. In these geographic areas water scarcity concerns are common. Rainwater harvesting has been used as a water conservation measure, particularly where other water resources are scarce. Harvested rainwater (HRW) is one possible alternative to address this global issue. National water quality standards for both potable and non-potable domestic usages are thus far undetermined as HRW is a quite new developing practice worldwide. Project Harvest (PH) is a citizen scientist driven program that teaches communities across the state of Arizona the scientific method. Over the course of three years, participants will collect rooftop HRW samples and send them to be analyzed for bacteria, organic and inorganic contaminants at University of Arizona by our team of scientists. PH seeks to fortify informal science learning in underserved communities and help generate water quality guidelines and recommendations for non-potable HRW domestic use. Specifically, we aim to investigate the presence of thirty target analyte chemicals in HRW by applying high-resolution liquid chromatography-tandem mass spectrometry (HR-LC-MS/MS). A subset of HRW samples were pre-concentrated using solid phase extraction (SPE) and an equal number were analyzed via direct injection (DI) to determine whether pre-concentration is necessary. Atrazine, 2,4-dichlorophenoxyacetic acid and Simazine were observed in pre-concentrated HRW samples. Chlorpyrifos, prometon, 2,4,5-trichlorophenoxyacetic acid, and glyphosate were non-detect in DI and pre-concentrated HRW samples. Preliminary results suggest the use of SPE as a necessary sample preparation technique for the analysis of HRW samples.

P-7 Impact of Radiant-Heat and Tempering Treatments on Decontamination of *Aspergillus* flavus on Shelled Corn

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Corn samples of 500 g at moisture content of 24% wet basis were inoculated with 1 mL of the spore suspension of freeze-dried *Aspergillus flavus*. After 5 days of incubation, samples were then heated using Infrared (IR) energy source equipped with catalytic IR emitters at product to emitter gap sizes 11, 22 and 36 cm, which translated to measured IR intensities of 6.90, 3.24 and 1.27 kW/m^2. The samples were heated for 30, 60, 90, 120, 150 and 210 seconds and tempered at a temperature of 70^{o}C for 4 hours. Treated corn was then placed in favorable conditions for mold growth (33^{o}C and 90% relative humidity) to examine the potential for mold regrowth after the treatments. Microbial loads (CFU/g-corn) were determined by plating treated corn on the selective media, Rose Bengal Agar (RBA) and incubated for

5 days in an incubator set at 33°C. Colony forming units were then determined by counting colonies on the incubated RBA plates. Aflatoxin concentrations (ppb) were determined using a fluorometric test procedure. Increasing IR intensity from 1.27 to 6.90 kW/m^2 resulted in considerable fungal load reduction. Additionally, increasing treatment duration and adding a tempering step caused statistically significant reductions in *A. flavus* and aflatoxin concentrations. Corn samples treated at the highest intensity and treatment duration had decreased capacity for fungal regrowth when placed in favorable conditions while aflatoxin concentrations remained unchanged. This work showed that IR heating and tempering of corn has the potential to significantly decontaminate microbes on corn and prevent their regrowth in storage.

P-8 Analysis of toxins and processing residues in commercial California-style olives

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California-style ripe olive are the most common type of table olive produced in the US. This processing method introduces several safety and quality concerns that are unique to CA-style olives. Styrene and acrylamide are neurotoxins found in CA-style black olives as by-products of processing, although the mechanisms of formation are unclear. Sodium benzoate and ferrous gluconate are processing aids with legal limits and adverse health effects at high concentrations. Concentrations of these toxins and processing residues have been scarcely studied in CA-style black olives, and have not been measured in newer products including green ripe and "natural" black ripe olives.
The goal of this project was to quantify toxins and residues in commercial CA-style olives and identify strategies to improve the safety and quality of these products. Analytical methods for measuring styrene, acrylamide, sodium benzoate and ferrous gluconate in CA-style olives using GC-MS, HPLC-DAD and ICP-MS were developed or adapted from previous work. Phenolics, which potentially affect toxin formation, were also measured using a newly-developed high throughput extraction method. These compounds were analyzed in forty commercial samples including both domestic and imported products, as well as black, green and "natural" olives. Processing aids were below legal limits in all commercial samples except one import. Imported products had higher styrene compared with domestic products. Green ripe olives had no detectable styrene and lower acrylamide concentrations. The results of this study can guide future research into formation mechanisms for these neurotoxins and be used to set quality standards for imported products.

P-9 Evaluation of Estrogenic Activity of novel Bisphenol-A alternatives, four Bisguaiacol-F compounds, by *in-vitro* assays

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Bisphenol A (BPA) has raised public interest due to the ubiquitous presence in environment, the increasing evidence on its estrogenic activity (EA) and adverse health effects. Bisguaiacol-F (BGF) is a lignin-inspired alternative to BPA using in the food contact materials. However, few experimental data are available to demonstrate the endocrine disruption potential of BGFs. In this study, EA of four BGF regioisomers at nine concentrations (from 10^{-13} to 10^{-5} M) was quantified as the relative maximum % 17-βestradiol (E2) (%RME2) by two sensitive and accurate *in-vitro* assays, human breast cancer cells MCF-7 cell proliferation assay and VM7Luc4E2 transactivation (TA) test. The MCF-7 cell proliferation assay revealed that the %RME2 of BGF1 (containing 92.9 mol% of *p, p*-BGF) and BGF4 (containing 30.1 mol% of *p, p'*-BGF and 62.0 mol% of *o, p'*-BGF) were 32.9% and 63.0% separately less than the %RME2 of BPA. BGF6 (containing 68.4 mol% of *p, p'*-BGF, 31.1 mol% of *m, p'*-BGF) and BGF8 (containing 75.7 mol% of *p, p'*-BGF, 23.9 mol% of *m, p'*-BGF) had no detectable EA at all test concentration. Similar EA of BGFs was obtained by VM7Luc4E2 TA test while there are several different dose-response due to the cytotoxicity of E2 and BPA on MCF-7 cells at high concentrations. All findings indicated both BGF6 and BGF8 are the potential less toxic alternatives to BPA. The findings first demonstrated that not all BGF molecules have little EA. Higher percentages of *m, p'*-BGF in the BGF6 and BGF8 might contribute to their undetectable EA.

P-10 Rapid GC-MS/MS Analysis of Chlorinated Pesticides in Animal Fat with SPE Clean-up

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Chlorinated pesticides have restricted use due to their toxicity and are highly regulated. The lipophilic disposition of chlorinated pesticides has rendered them prone to bioaccumulation in fatty tissues thus resulting in difficulty with excretion from the body. Common extraction methodology for chlorinated pesticides in fat tissues, such as FDA Pesticide Analysis Manual 304, is laborious, uses large volumes of solvents and is subject to high variability in extraction efficiency. A method has been developed and validated for the analysis of chlorinated pesticides in animal fat using solid phase extraction. This method reduces preparation time of extraction by at least two fold.

P-11 Rapid Antemortem Detection of Tetracycline Residues in Porcine Oral Fluids

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Based on 2016 antibiotic farm usage records, tetracyclines are the most common class of antibiotics used in swine production. To detect and quantitate tetracyclines in porcine muscle, post-mortem samples have typically been sent to analytical laboratories for HPLC or LCMS determination which can require a few days for results and significant expense. Previous literature has shown that oxytetracycline concentration in oral fluids matched closely with drug levels in muscle. A rapid and inexpensive ante-mortem screening test for tetracyclines in oral fluid could therefore provide a rapid estimation of drug levels present in tissue, decrease analysis time and reduce testing expense. We have developed a diagnostic method to screen porcine oral fluid for tetracyclines using lateral flow devices that provides results within minutes. Oral fluid samples spiked with 50 ppb doxycycline carried through the sample preparation process showed visually positive results using the lateral flow devices. Direct sampling of spiked oral fluid samples showed better sensitivity for tetracycline, chlortetracycline and oxytetracycline and visual detection for those at 50 ppb is being confirmed. These devices were originally designed to screen milk for several drug residues. This work shows the devices and sampling procedure have now been extended to detect tetracyclines in oral fluid samples collected from chew ropes providing a rapid and cost-effective screening method for tetracycline residues in swine.

P-12 Comparing GC-ECD/NPD and GC-MS/MS on analyzing Chicken and Egg

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In 2017, some insecticide were detected on eggs and chickens in Korea. And some insecticide were over the MRM. For the faster analysis, this research compared GC-ECD/NPD and GC-MS/MS on chicken meat and eggs to evaluate the better performance. Agilent 7890B was used for GC-ECD/NPD. For the MS/MS analysis, Shimadzu TQ8040 was used. On pretreatment, modified QuEChERS method was used and both are very similar but show some difference on sample weight and extraction solvent. On GC-ECD/NPD 20 g of chicken and egg were used and 50 ml/20 ml(chicken/egg) extraction solvent were used. Also compounds were divided into 4 groups, 2 for ECD and 2 for NPD. On GC-MS/MS, 10 g of samples were used and 50 mL of extraction solvent were used. All compounds were in the one group. 64 compounds were compared for linearity, LOQ(limit of quantitation) and recovery including RSD(relative standard deviation). On linearity, most of the compounds showed over 0.99 linearity both on GC-ECD/NPD and GC-MS/MS. 55 compound's LOQ was under 0.05 mg/kg on GC-ECD/NPD while 64 compound's LOQ was under 0.05 mg/kg on GC-MS/MS. For the recovery test 0.005 mg/kg and 0.05 mg/kg were used. Acceptable recovery range was 70-120% and RSD was under 20%. On GC-ECD/NPDNPD o 55 compounds met required condition on chicken and 63 compounds contented required condition on egg. For the GC-MS/MS, 56 compounds met required condition on chicken and 55 compounds met required condition.

P-13 Rapid Determination of Hypoglycin A in Ackee by Liquid chromatography/Tandem mass spectrometry

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A simple liquid chromatography/tandem mass spectrometry (LC-MS/MS) analytical method was developed for the determination of toxic amino acid Hypoglycin A in ackee fruit. This free amino acid was retained on a mixed-mode column without the need of pre-column derivatization. A 3-g test portion was shaken with ethanolic solution. After centrifugation, the sample extract was diluted and injected and analyzed within 15 min by LC-MS/MS. Two MS-MS transitions were monitored in the method for the compound to achieve true positive identification. An isotopically-labeled internal standard of L-Leucine d3 was used to correct for matrix effect and/or instrument signal drift. The average recovery for all analytes at 17, 33, and 66 µg/g (n = 18) ranged from 70-120%, with a relative standard deviation of ≤ 20%.

P-14 Rapid determination of polar pesticides and plant growth regulators in fruits and vegetable by LC/MS

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This paper explains a quick extraction procedure to recover very polar compounds from apples, carrots, and sugar snap beans. The new mixed-phase HPLC column allows direct determination of polar analytes of pesticides and plant growth regulator including amitrole, chlormequat, mepiquat, cyromazine, ETU, PTU, perchlorate, and daminozide without the use of time-consuming derivatization steps, HILIC, or ion-paring reagents. A mass spectrometer with electrospray in positive/negative mode is used to detect and confirm the presence of these analytes. Eight isotopically-labeled internal standards corresponding to each analyte were used to correct for matrix suppression effect and instrument signal drift. The average recovery for all analytes at 20, 40, and 250 ng/g (n = 6) ranged from 73-136%, with a relative standard deviation of ≤ 20%. The proposed method is more rugged than other direct determination methods and is the only LC/MS method currently available for the determination of these analytes in a single run.

P-15 Metabolite profiles of HepG2 human hepatocarcinoma cells reveal different responses to the insecticide permethrin and its metabolite, 3-phenoxybenzoic acid

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Permethrin is a widely used neurotoxic insecticide, primarily for parasite control. Due to its widespread use, humans are frequently exposed to this compound, although it is generally considered to be safe. However, several rodent studies have demonstrated potential hepatotoxicity, prompting concerns over its potential effect on humans. The liver is the primary site of metabolism for permethrin, where is it converted to 3-phenoxybenzoic acid (3-PBA) before glucuronidation and excretion. This study aimed to characterize the changes that occurred in liver cells in response to both permethrin and 3-PBA.

This study exposed HepG2 human hepatocarcinoma cells to permethrin or 3-PBA for 48 hours and analysed the intracellular metabolites using an Agilent 7200 GC-QToF-MS. Resulting data was modeled using principal component analysis, and showed a clear difference in metabolite profile between control and permethrin-exposed cells. Cells exposed to 3-PBA clustered between the other two groups, suggesting an intermediate metabolite profile. Data demonstrated that the key changes resulting from exposure to permethrin were increased anaerobic glycolysis, oxidative stress leading to mitochondrial dysfunction and increased lipogenesis and nucleotide biosynthesis. Changes observed on exposure to 3-PBA were similar, without an increase in anaerobic glycolysis, suggesting that the impact on metabolism was reduced compared to permethrin. Overall, the study demonstrates that metabolic alterations occur in HepG2 liver cells in response to both permethrin and its metabolite, 3-PBA, but that the latter causes less severe changes to the biochemistry of the cell.

P-16 Metabolite profiles of B50 rat neuroblastoma cells change across multiple passages

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The drive to reduce the use of animals in toxicology studies has led to a renewed interest in the suitability of in vitro models based on cultured cells from various animal species. Increasingly, these models are being combined with 'omics techniques such as metabolomics. Immortalised cell lines represent a convenient system for such studies, but there are numerous factors that need to be taken into consideration when interpreting data. Depending on the generation time of the specific cell line, cells will need to be passaged, or 'split', every few days. To date, there have been no studies examining changes in the metabolite profile across multiple passages, so little is known about the comparability of data between passages. This study sampled B50 rat neuroblastoma cells across 15 consecutive passages and analysed the intracellular metabolites using a Shimadzu QP-2010 Ultra GC-MS. Resulting data was modeled using principal component analysis. While changes from one passage to the next were relatively subtle, a clear shift could be observed in the metabolite profile across the 15 passages, with 'early' passages (e.g. 1-5) clearly distinct from 'later' ones (e.g. 11-15). Early passages showed differences in carbohydrates and fatty acids, while variation in later passages was largely based on inositols. Overall, this study demonstrates that considerable care must be taken in experimental design to ensure that data from toxicology studies are comparable when conducted across more than one passage.

P-17 Multi-Residue Analysis of Pesticides in Medical Cannabis

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Significant attention has been given to the safety of medical cannabis as Canada prepares to legalize recreational cannabis. With the discovery of unauthorized pesticides in cannabis products in other jurisdictions, there was an urgent need to develop reliable methods of analysis in Canada. As cannabis is a controlled substance, there is little precedent in pesticide method development, making compliance-driven laboratory analysis challenging. This work describes the development and implementation of robust, validated and accredited methods for the analysis of pesticide residues in cannabis. Three multi-residue methods of 85 pesticides in cannabis leaves, flowers and oil were developed by HPLC-MS/MS, GC-MS/MS and GC-MS. Validation at spiking levels as low as 0.01 µg/g showed recoveries within 70-130%. These methods were the first to be accredited in Canada and were successfully utilized in unannounced inspections of Canadian licensed producers to ensure medical cannabis products were in compliance with Canadian pesticide regulations.

P-18 Survey of Mycotoxin Residues in Oregon Cannabis Crops by LC-MS/MS

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Cannabis is being sold in many states for both recreational and medical applications but testing for the presence of highly toxic fungal metabolites is rarely required by state regulations. Mycotoxins can be harmful to human health at very low concentrations which require powerful instrumentation to detect and measure accurately. Using the combined resolution power of liquid chromatography and tandem mass spectrometry, cannabis flower samples from a variety of producers across the state of Oregon were analyzed for eleven different mycotoxins. The results of this survey will indicate whether stricter testing regulation should be established to ensure consumers and patients have access to clean and safe cannabis products.

P-19 Optimization of Sample Preparation for Pesticide Analysis in Oil-Based Cannabis and Hemp Products

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Popular oil-based cannabis and hemp products like CBD oil are composed primarily of food grade oils such as coconut derived medium chain triglycerides (MCT) oil. These high fat products are difficult matrices for trace level pesticide residue analysis. This work optimizes MCTs and lipid removal so that acceptable detectability and data quality for pesticide analysis can be achieved. A pass through SPE sample preparation product designed for high capacity lipid removal, LipiFiltr™, was used to minimize matrix coextractives. Pesticides regulated for cannabis in Oregon were tested for recovery. Several combinations of dilution solvents and dilution factors were tested with the LipiFiltr™ push through purification cartridge. Each was evaluated by gravimetric analysis, volatile co-extractives, and lipid removal via gas chromatography-mass spectrometry. Pesticide recovery and reproducibility were evaluated by liquid chromatography-mass spectrometry.

P-20 Screening of pesticides and mycotoxins in Apple Ciders by Liquid Chromatography-High Resolution Mass Spectrometry

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Based on the trend of increasing consumption of apple ciders especially from craft cider houses in the Czech Republic, we designed an experiment which provides information about levels of pesticides and mycotoxins residues in these kind of beverages. The Main target of this experiment was not only to determine the concentrations of these residues coming from apple peels into the final product but also to evaluate their potential risk to consumer's health. Samples were chosen mainly from local ciders as well as from several foreign ciders. Also, a few organic ciders were involved in the experiment in order to confirm the declaration.
In this study, we used QuEChERS based extraction method in combination with column SPE addition sample clean-up to achieve the lowest detection limit possible due to low concentrations of pesticide residues in the samples. The samples were analyzed using a high-resolution, accurate-mass (HR/AM) Q-Exactive instrument and this applied method made quantification of pesticide residues in ciders possible at 0.2 ppb level for most of fifty target pesticides. Additionally non-target screening mode enabled to verify presence of over 300 pesticides using comprehensive library including names, retention time and empirical formula as well as verified fragments.

P-21 Determination of Glyphosate – AMPA and Glufosinate in various food matrices without derivatization with molecularly imprinted polymers (MIP) SPE cleanup

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Glyphosate and Glufosinate are closely structured herbicides referred to as phospho-herbicides. Glyphosate is the most commonly used herbicide worldwide with around 25% of the global herbicide market. It undergoes rapid microbial degradation in plants, soil and water to the metabolite aminomethylphosphonic acid (AMPA). Codex alimentarius has defined a MRL (maximum residue limit) for Glyphosate of 0.05mg/Kg in meat or milk and 30mg/Kg in cereals and for Glufosinate, 2mg/kg of soybean.
As very polar molecules, the analysis of these molecules is still a challenge. Indeed, they are difficult to extract with organic solvents and common solid phase extraction (SPE) sorbents. A new SPE sorbent based on Molecularly Imprinted Polymers (MIP) was developed for these products. A MIP is a synthetic material with artificially generated three-dimensional network which shows affinity for a target molecule. Based on this technology, a new and powerful SPE clean-up method was developed for the analysis of Glyphosate, AMPA and Glufosinate in several food matrices such as cereals. The analysis does not require derivatization of these compounds. SPE results have shown a good ability of the sorbent to catch these molecules with good recoveries. This sorbent has also been tested on urine.

P-22 Determination of contaminants residues (Glyphosate, mycotoxins) in cannabis with molecularly imprinted Polymers (MIP) SPE cleanup

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In some USA states and several countries in the world, the sale and possession of cannabis is now legal for both medical and recreational use. These new regulations open the door to the legal consumption of a new product. Consumers must expect a safe product and laboratories must develop analytical methods to analyze potential contaminants residues presents such as pesticides or mycotoxins in this new matrix.
In this poster, molecularly imprinted polymers for the analysis of contaminant residues in cannabis like plants have been used as a clean-up. A protocol was developed for the analysis of glyphosate, AMPA and glufosinate as well as mycotoxins such as Ochratoxin A.
A MIP is a synthetic material with artificially generated three-dimensional network which shows affinity for a target molecule. Based on this technology, a new and powerful SPE clean-up method was developed for the analysis of pesticides and mycotoxins in cannabis. SPE results have shown a good ability of the sorbents to catch these molecules with good recoveries.

P-23 Automated Preparation of Beverages for the Analysis of Bisphenol A and Selected Analogues

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Bisphenol A (BPA) is a high commercial use, chemical building block in the production of polycarbonate plastics and epoxy resins. Recently, BPA has come under scrutiny for its widespread prevalence in human exposure monitoring studies and food and beverage testing. Due to the potential endocrine disruptive nature of BPA, restrictions of its use in food contact packaging are growing. Alternative BPA-analogues are now being used to produce "BPA-free" products, however, these replacement compounds are now making their way into the food chain in a similar fashion as the parent, and may have the same adverse hormonal effects.
Bisphenol A, as well as the analogs bisphenol B, F, S, Z AF and AP were monitored in several beverage types sold in plastic bottles, and warm water extracts of polycarbonate bottles and toys. Using the PrepLinc Large-Volume Injection (LVi) system allowed large volumes of aqueous sample matrix to pass through commercially available SPE cartridges and disks automatically. The autosampler will accommodate a variety of sample bottle sizes, therefore sample is taken directly from the container used to dilute the aqueous sample or a bottle containing the warm water extract. In this study, the labor-intensive steps of SPE conditioning, loading, elution and concentration were reduced to two steps: 1) loading samples on to the instrument; and 2) programming the sequence with saved method parameters. This provided for a fast and simple automated method for the manual process of loading 1 liter of water to an SPE, subsequent elution, and concentration for HPLC analysis.

P-24 Improvements to Targeted & Untargeted Pesticide Residue Analysis: Fast and Flexible Analyte Finding for GC/MS and GCxGC/MS.

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In recent years there has been a dramatic expansion in the number of pesticides utilized in food products, especially in emerging markets and commodities. With this expansion, analytical techniques with the ability to acquire comprehensive non-targeted data have become increasingly important. However, large lists of target compounds can be challenging and time-consuming to maintain, often requiring multiple standard and sample injections in order to develop methods for different matrix interferences and analytical conditions. This presentation will showcase the creation and utilization of a target list of pesticides using software tools designed to make processing comprehensive data faster, easier, and more effective. With enhanced flexibility in data processing parameters, fewer injections are needed to fully develop an easy-to-update, automated data processing method for targeted analytes. A proposed target list containing over 200 commonly used pesticides serves as a starting point for the development of a new pesticide analysis solution kit. The ability to easily add emerging pesticides to the stock list will also be demonstrated. This workflow utilizes a robust benchtop time-of-flight GC/MS system capable of femtogram level sensitivity and is expected to compete

favorably with current workflows employing systems requiring selected ion monitoring (SIM) or MS/MS to achieve desired sensitivities.

P-25 Separation of Mycotoxins by UHPLC-MS/MS using a Novel C18-based Stationary Phase

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Mycotoxins are a group of naturally occurring chemicals produced by certain molds. They can grow on a variety of different crops and foodstuffs including cereals, nuts, spices, dried fruits, apple juice and coffee, often under warm and humid conditions. Mycotoxins are considered one of the most important contaminants of agricultural commodities, both in the field and in storage.
Aflatoxins - in particular aflatoxin B1, are genotoxic and carcinogenic and may cause liver cancer in humans, whilst ochratoxin A and the trichothecenes HT-2 and T-2 can cause various toxic effects. Due to their potential toxicity at low levels to both humans and animals, monitoring and control of certain mycotoxins is important within the food industry. This poster shows a UHPLC-MS/MS separation seven of the most concerning mycotoxins from a food safety perspective include the aflatoxins (B1, B2, G1, G2), ochratoxin A, and toxins produced by Fusarium molds, including trichothecenes (T-2 and HT-2 toxins). The poster also explores the importance of selectivity and stationary phase chemistry. Separation of all analytes was acheived on a novel ACE C18-AR column incorporating enhanced aromatic selectivity in less than 5 minutes. The low bleed characteristic of this phase make it ideal for use with tandem MS detection, permitting low level detection and identification of these key components.

P-26 Isomer Ratios of Pesticides for the Cannabis Industry

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The State of California recently legalized marijuana for recreational use. The regulations include setting limits for acceptable pesticides as well as reporting isomer ratios for applicable pesticides. These pesticides may be mixtures of various isomers or similar compounds. Isomers can be structural isomers (different bond locations) or stereoisomers (resulting from unsaturation (cis- and trans-) or chiral centers (R- or S-). Determining isomer ratios may be difficult, method specific, vendor specific, and even lot number specific. This study shows the types of uncertainty associated with reporting isomer ratios.

P-27 The Challenge of Designing a Master LC/MS Calibration Solution for Pesticide Solution Product Verification

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As a custom reference material manufacturer, we produce approximately 400 solutions weekly with about 25% of those solutions being made for the first time. This places a high demand on generating excellent data quality and high throughput in the quality control laboratory. To facilitate both of these aspects, we have developed a LC/HRAMS pesticide calibration method containing approximately 800 analytes. Stability data, solvent, pH, and chemistries will be discussed.

P-28 Trace Level Pesticide Detection Utilizing Gold Nanoparticles and Portable, Handheld Surface Enhanced Raman Spectroscopy

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Pesticides play a critical role in protecting food crops from insects, fungi, weeds, and other unwanted pests. The increasing use of these pesticides to maintain food production and quality leads to potentially dangerous residues

remaining on the food products. A rapid and non-destructive technique for trace level detection of pesticides at parts-per-million (ppm) or parts-per-billion (ppb) is surface enhanced Raman spectroscopy (SERS). A key feature of SERS is that it utilizes noble metal nanostructures to increase the weak Raman signals from analytes. We present a novel SERS substrate involving gold nanoparticles suspended in water that can be used to help identify four different pesticides: thiram, malathion, captan, and phosmet. In order to observe the desired Raman spectral signatures of these pesticides, apple skin contaminated with each chemical was swabbed and added to the colloidal gold nanoparticle suspension followed by interrogation with 785 nm laser excitation and a handheld, portable Raman instrument. This current technique can detect each of these pesticides down to 1 part per million, where the pesticide residue tolerances on apples as established by the 2018 Code of Federal Regulations for thiram, malathion, captan, and phosmet are 5, 8, 25, and 10 parts per million, respectively. The results presented here indicate that SERS is a useful tool for identifying pesticide residues on the surface of fruits for food quality and safety control.

P-29 High sensitivity analysis of pesticides in botanical dietary supplements

Jeffrey Dahl and Tairo Ogura

Shimadzu Scientific Instruments, Columbia, Maryland

Dietary supplements are widely used but their raw materials are subjected to fewer regulatory controls than staple foods. To ensure quality the US FDA requires identity and quality testing, but most botanicals do not have specific regulations. To analyze complex botanicals for residual chemicals such as pesticides, LC-MS-MS is needed for high sensitivity, high confidence results. We developed an LCMS method with improved detection sensitivity for chemical residues in botanicals.
Representative samples of botanicals were homogenized by grinding and extracted with acetonitrile accompanied by shaking and sonication. Depending on the complexity of the matrix, samples were additionally cleaned up using dispersive solid phase extraction to remove unwanted matrix components. Analysis was carried out by LC-MS-MS using a triple quadrupole mass spectrometer.
Method performance was validated and a sample of randomly selected botanicals purchased from online retailers was analyzed. Using our newly developed method, we are able to characterize the extent of residual pesticides present in popular botanicals.

P-30 Determination of Organochlorine Pesticides and Polychlorinated Biphenyls Using GC-MS/MS Operated in the MRM Mode

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The determination of chlorinated pesticides (OCPs) and polychlorinated biphenyls (PCBs) in environmental matrices are common analyses in most environmental laboratories. These compounds are typically analyzed by employing solid phase or liquid liquid extraction with Methylene chloride, concentration, solvent exchange into hexane, and interference removal using acid, copper, or column chromatography. Analysis is done using gas chromatography (GC) with electron capture detection (ECD) and requires confirmation of every detected component on another, dissimilar GC column. GC-ECD techniques are prone to positive and negative bias in complex matrices resulting in unnecessary cleanup costs and/or violations of NPDES permits. Clearly, a new method for pesticides and PCBs, based on modern GCMS technology is needed. This poster describes use of a tandem GC-MS/MS method using Multiple Reaction Monitoring (MRM) mode for sensitive and selective detection and quantitation of organochlorine pesticides and PCBs. A database with optimized MRM transitions for all of the OCPs and PCBs including relative retention times for all components makes method setup possible within minutes. The use of GCMSMS MRM mode provides enhanced selectivity, specificity and sensitivity in complex matrices with potential co-eluting interferences.
This poster presents all instrument operating conditions, and instrument method performance statistics including method linearity, accuracy, precision, and instrument detection limits for all compounds.

P-31 Simultaneous Quantitative Analysis of Pesticide and Mycotoxin Residues in Cannabis Oils

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Methods determining the presence and quantitative amount of pesticides in cannabis oils have become important tools in qualifying the safety of patients as regulation for the use of medical marijuana becomes more prevalent throughout the United States. In addition to pesticide analysis, the presence and quantitative amounts of mycotoxin residues in these oils due to contamination from molds and other fungi is needed to ensure that these poisonous carcinogens are not present in the medications prescribed to patients. Most existing methods utilize long separation times on column and separate the analysis of pesticides and mycotoxins into multiple methods. These issues are resolved with the use of a Shimadzu Nexera series UHPLC system coupled to a Shimadzu 8060 triple quadrupole mass spectrometer. Simultaneous analysis of compounds in positive and negative mode, ultra-fast polarity switching, and ultrafast-scanning speeds are shown here to allow for accurate, quantitative, and highly selective analysis of a series of 55 pesticides and 5 mycotoxins in one method. Multiple calibration curves and QC's were prepared and run to demonstrate percent accuracy and RSD values <20%. All calibration curves are linear with R^2 values >0.990. The method is highly selective, highly automated, robust and achieves linearity over the extent of the dynamic range for each compound.

P-32 Supercritical Fluid Extraction of Pesticides in Fish Tissue

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There are a number of techniques used for the extraction of pesticides from fish tissue. These extractions often use a large amount of organic solvent, required extensive sample preparation, or are performed under conditions that may degrade certain pesticides. Supercritical fluid extraction was evaluated for the recovery of twenty organochlorine pesticides from fish tissue using a 100% carbon dioxide extraction phase. The pesticides were trapped on a C18 column and eluted with hexane for offline analysis by GC-MS or GC-ECD. Recoveries of the pesticides ranged from 40-105%, however 16 pesticides could be recovered at levels above 80%. Details of the analysis and the results of additional parameter changes to increase extraction efficiency will be presented. The automated extraction of up to 48 samples can be performed with this technique.

P-33 "Not Your Kid's Apple Juice": An examination of Arsenic Content in American and European Hard Ciders

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Alcoholic hard ciders have a long history around the world but only have become readily available in the United States over the past few decades. Over the last several years, several studies have been conducted showing the presence of arsenic in apple juices and wine. Arsenic based pesticides, particularly lead arsenate, were in widespread and common use in the United States up until the 1970's until its final ban in 1988. Despite arsenic residue being recognized as a potential problem from the turn of the century, lead arsenate was one of the most widely used pesticides in the nation and was applied to millions of acres of crops through the 1940's. Lead arsenate was the most commonly applied pesticide in apple orchards and many of the historical apple orchards around the world still produce apples and apple products, and so potential for contamination of arsenic compounds remain. Heavy metal pesticides were designed to be persistent and this issue can cause environmental and health problems decades after the pesticides were banned. In this study, samples were obtained of popular American and European hard ciders. Modern hard ciders are produced from either fresh apples or apple concentrates. American ciders are required to be at least 50% apples or concentrate, while UK ciders are required to be at least 35% apples. Samples were digested using microwave digestion and testing by ICP-MS and LC-ICPMS to determine total arsenic content and potential content of different arsenic species.

P-34 Rapid Extraction, Cleanup and Determination of Multiple Pesticide Residues in Difficult Matrices Utilizing Energized Dispersive Extraction and UPLC MS/MS

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In the modern world, consumers increasingly want to know what is in their food and that the substances they are putting in their body are safe. This, along with stringent regulatory requirements, is leading the call for improved extraction of food contaminants such as pesticides. The QuEChERS method has been shown to be practical for pesticide analysis on a number of different sample types and is increasingly being employed on more difficult matrices. Unfortunately, some matrices by either their nature or their economic value can be difficult to analyze with just the QuEChERS method alone and show lower recoveries of the pesticides than are often observed with matrices that are less complex. While the QuEChERS method is relatively quick compared to other methods, it is also a manual and tedious process that remains the bottleneck. A new technology for sample preparation that will eliminate the bottleneck and improve pesticide recoveries for difficult food matrices is being reviewed. Herein this new sample preparation technology, Energized Dispersive Extraction, paired with UPLC MS/MS analysis that allows for extraction, cleanup, filtration and analysis in less than fifteen minutes of difficult matrices is presented. Energized Dispersive Extraction combines the extraction and cleanup process in one-step and is applicable to many different types of matrices such avocado and hops. The extraction and determination of multiple pesticide residues from these difficult matrices will be described.

P-35 Investigation of Garlic and Onion Containing Food Products for a False Positive Sulfite Response by LC-MS/MS

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Sulfites are a family of additives regulated for use in food products. In the US, sulfites must be declared on the label if they are present in concentrations greater than 10 mg/kg (determined as) SO2 because an allergic-like response has been reported in a small subset of the population upon consumption of sulfite-containing products. The current US regulatory method for sulfites, the optimized Monier-Williams (OMW), produces false positive results with vegetables from the *Allium* (garlic) and *Brassica* (cabbage) genera due to extraction conditions that are thought to cause endogenous sulfur compounds to release SO_2. Recently, an LC-MS/MS method was developed for sulfites but has only been tested with samples that are 100% *Allium* or *Brassica*. To get data on more representative products, three blank matrices: chips, hummus, and quinoa were spiked with concentrations of an onion and garlic powder. It was determined that even at concentrations of 8% garlic or onion, the measured sulfite concentration was below the 10 ppm SO_2 labeling threshold. Higher concentrations of powders were tested to determine how much would need to be added to a matrix to produce the concentrations that would result in a false positive response. Commercial dried garlics were evaluated to determine the variation in responses that could be encountered in future reglatory samples. Recovery studies were conducted to determine if these methods would detect added sulfite. The ability to eliminate false positives will result in a greater reliability in the accurate determination of added sulfite to ensure compliance with labeling requirements.

P-36 Mass-Spectrometric Separation and Quantification of Permethrin and Cypermethrin Residues on Vegetables Grown under Greenhouse Conditions

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An analytical procedure was developed to quantify permethrin [3-Phenoxybenzyl (1RS)-cis, trans- 3-(2, 2-dichlorovinyl) -2, 2-dimethylcyclopropanecarboxylate] and cypermethrin [cyano-(3-phenoxyphenyl) methyl] 3-(2, 2-dichloroethenyl)-2, 2-dimethylcyclopropane-1-carboxylate] residues on treated beans and cucumber leaves and fruits. Fruits and leaves were collected at different time intervals of 1 h to 25 d following spraying of a mixed formulation of the two pyrethroids insecticides to determine their dissipation constants (K values) and half-lives ($T_{1/2}$ values). A simultaneous extraction procedure was carried out using hexane and plant extracts were cleaned-up using a 1.2 × 2 mm i.d. open glass chromatographic column packed with Florisil. A gas-chromatograph (GC) was used for quantification of individual

pyrethroid insecticide using an electron capture detector (GC-ECD). Residues of the two pyrethroids were confirmed using a GC equipped with a mass selective detector (GC-MSD) in total ion mode. The GC mass spectra revealed the presence of permethrin isomers at retention times of 26 and 26.6 min that correspond to the *cis*- and *trans*-isomers, respectively. The GC mass spectra also revealed the presence of cyperpermethrin isomers at retention times of 30.3, 30.9, 31.3, and 31.5 min that correspond to its four main isomers. The initial total residues were greater on the leaves than on fruits. The electron impact mass spectrum of permethrin extracted from cucumber and bean leaves and fruits indicated the molecular weight of 390 and molecular ions of m/z 183, 163, 127, 91, and 51, along with other characteristic fragments ions that corresponds to permethrin ion fragments.

P-37 Development and validation of a method for Polyoxin B and Polyoxin D analysis in fruits using LC-MS/MS

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Polyoxin B and polyoxin D inhibit the biosynthesis of chitin as fungicide. Several analytical methods have been reported for its determination. Microbiological assay had been used. However, this method is not suitable to determine residues exactly in agricultural products and food. In addition, it took lots of time and process is very complicated. A highly sensitive liquid chromatography-tandem mass spectrometry (LC-MS/MS) method was developed for analysis of Polyoxin B and polyoxin D. Stock solution was prepared by dissolving in 50% methanol and was diluted further with methanol to the required concentration before use. Extraction of Polyoxin B and polyoxin D from matrices was performed using a QuPPe method. A portion of the sample was placed in a polypropylene centrifuge tube, and 1% formic acid in Methanol was added and the centrifuge tube was shaken for 15min by a shaker vigorously. And centrifuge for 5 minutes at 3500 rpm to obtain the extract. The supernatant was passed through a PTFE filter and injected to LC-MS/MS. Performance evaluation was carried out on apple, tomato, pear. All commodities spiked with two concentration levels of 0.01 and 0.1 mg/kg. The recoveries were within 71.8 ~ 110.6 % with RSD of ≤4.3% and the limit of quantification(LOQ) of method were ≤ 0.006 mg/kg in all commodities. Linear calibration functions with correlation coefficients were obtained R2>0.995.

P-38 An Efficient Testing System using UHPLC Q-Orbitrap Mass Spectrometry for the Analysis of a Large Number of Pesticides in Fruits and Vegetables

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Analysis of pesticide residues in fruits and vegetables has been an ongoing challenge for the Canadian Food Inspection Agency (CFIA). Historically, single analyte or single class methods were used to determine pesticides; later, multi-residue methods were developed to streamline resources. Unfortunately for the quantitative analysis of pesticides, a full calibration standards suite is required. To mitigate the amount resources spent on standard preparation, standard costs and data processing time, the CFIA Calgary Laboratory R&D Unit has developed two multi-pesticide methods, a screening and a quantitative, for fruits and vegetables that complement each other. These methods use UHPLC-ESI with Q-Orbitrap high resolution mass spectrometer in Full Scan MS or Full MS/DIA (data independent acquisition) mode.
This poster presents workflow examples of Q-Orbitrap applications for target screening and quantification. A semi-automated screening method incorporated an in-house compound database containing ~450 pesticide entries was developed and validated. The purpose of the screening method was to save time and resources. The combination of software processing (TraceFinder 3.3) and in-house compound database alleviated the need for a full standard mix. This also allowed screening of uncommonly used pesticides to be done economically. The multi-residue quantitative method was developed and validated to determine ~450 pesticides. This method would be used to quantitate the positive screening results. This poster presents the process of the development, validation, challenges and results for both the screening and the quantitation methods. The combination of both methods formed a powerful and efficient system to analyse pesticide residues in fruits and vegetables.

P-39 A Novel, Resource Saving Screening Method for ~850 Pesticides Fruits and Vegetables using Ultra High Performance Liquid Chromatography coupled with Q-Orbitrap Mass Spectrometer (UHPLC/ESI-Q-Orbitrap MS)

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With advanced technology, such as UHPLC and Q-Orbitrap high resolution mass spectrometry, multi-residue pesticide analysis methods are routinely developed. These methods typically require the preparation of complicated analytical standards, and the data processing is very intensive and time consuming. Historically, screening methods have been used to mitigate these issues, but were only effective for methods with a relatively small number of compounds and would still require the preparation of a full suite of reference standards. This poster presents a validated method that can screen up to ~850 pesticides in a single injection 14 minutes run, and how it can significantly save laboratory resources.
The screening method was validated according to from SANTE/11945/2015 guidelines. A modified QuEChERS extraction was used to extract pesticides from fruit and vegetable matrices. Extract were run by Ultra high performance liquid chromatography (UPLC) in combination with Q-Orbitap mass spectrometry. Data processing was via proprietary software (TraceFinder 3.3), which integrates an in-house developed compound database (CDB). This combination allowed the software to use the compound database of 850 pesticides as a reference for screening, thus allowing for the semi-automated data processing of sample batches, without the need to prepare a full suite of standards. The validated method is able to screen 761 pesticides at 10 ppb and 820 pesticides at 100 ppb in sample. The combination of an efficient extraction process, capable instrumentation, and semi-automated data processing resulted in significant savings in money, time, and laboratory resources.

P-40 Mercury Cumulative Density Distributions in Finfish and the Implications for Risk Assessment

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Seven different retail finfish; grouper, mahi, swordfish, snapper, orange roughy, canned light tuna and canned white tuna were analyzed for mercury by ICP MS. The cumulative density function of the mercury concentrations was analyzed for "best" fit using *fitdisplus* package in R. The Akaike Information Criterion was used to select the distribution that best fit the data set. For all fish except canned white tuna a gamma distribution was determined to be the best fit. Canned white tuna had a normal distribution. Monte Carlo simulations were performed to determine the mercury concentrations at percentiles of the cumulative density function 0.1, 0.5, and 0.9. The mercury concentrations determined by cumulative density function at the percentile 0.5 was compared to the empirical mean to evaluate differences in the "most likely" exposure level.

P-41 Determination of aromatic amines and trace organic constituents in D&C Red No. 33 using LC-MS/MS

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D&C Red No. 33 (R33, disodium salt of 5-amino-4-hydroxy-3-(phenylazo)-2,7-naphthalenedisulfonic acid) is a color additive permitted for use in drugs and cosmetics. R33 is batch certified by FDA to ensure compliance with regulatory requirements that include ppb-level specifications for four aromatic amines (4-aminoazobenzene, 4-aminobiphenyl, aniline, and benzidine) and two additional trace organic constituents (azobenzene and 1,3-diphenyltriazene). We have developed and validated a new ultra-high performance liquid chromatography and tandem mass spectrometry (LC-MS/MS) method for directly determining these six analytes following extraction using the QuEChERS ("Quick, Easy, Cheap, Effective, Rugged, and Safe") technique. The new method also may be used to screen for 4-aminobenzonitrile, 2-aminobiphenyl, and *p*-toluidine as potential good manufacturing practice violations. Currently, the aromatic amines

are determined by a labor-intensive method that includes chloroform extraction, derivatization, and analysis of the reaction products by reversed-phase high-performance liquid chromatography. The trace organic constituents are determined separately by a similar method. The new method is more sensitive than the current methods and does not use chloroform, and the ready-to-use extraction kits make our method faster and less labor intensive. We used our new method to analyze samples from certified R33 batches from domestic and foreign manufacturers that requested certification during the past five years. Results for the analytes obtained by the new method and current methods will be presented.

P-42 Determination of unsulfonated aromatic amines in the color additives FD&C Yellow No. 5 and FD&C Yellow No. 6 using LC-MS/MS

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FD&C Yellow No. 5 (Y5, trisodium salt of 4,5-dihydro-5-oxo-1-(4-sulfophenyl)-4-[4-sulfophenyl-azo]-1H-pyrazole-3-carboxylic acid) and FD&C Yellow No. 6 (Y6, disodium salt of 6-hydroxy-5-[(4-sulfophenyl)azo]-2-naphthalenesulfonic acid) are color additives permitted for use in foods, drugs, and cosmetics. Y5 and Y6 are batch certified by the U.S. Food and Drug Administration to ensure compliance with regulatory requirements that include ppb-level specifications for aniline, benzidine, 4-aminobiphenyl, and 4-aminoazobenzene. We have developed and validated a new liquid chromatography-mass spectrometry (LC-MS/MS) method for directly determining the four unsulfonated aromatic amines following extraction using the QuEChERS ("Quick, Easy, Cheap, Effective, Rugged, and Safe") technique. Currently, the analytes are determined by a labor-intensive method that includes chloroform extraction, diazotization and coupling with 4,5-dihydro-5-oxo-1-(4-sulfophenyl)-1H-pyrazole-3-carboxylic acid, and analysis of the coupling products by reversed-phase high-performance liquid chromatography. Our new LC-MS/MS method is more sensitive than the current method and the ready-to-use extraction kits make our method faster and less labor intensive. The same method can also be used to screen for two other commonly found unsulfonated aromatic amine impurities, 2-aminobiphenyl and 4-aminobenzonitrile. We used our new method to analyze samples from certified Y5 and Y6 batches from domestic and foreign manufacturers that requested certification during the past five years. Results for the six unsulfonated aromatic amines obtained by the new method and the current method will be presented.

P-43 Performance of a comprehensive multi-class veterinary drug analytical method in a variety of animal matrices on a new best-in-class triple quadrupole mass spectrometer

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Veterinary drugs are broadly defined as chemicals that are used to protect animals from contracting disease, promote growth, and in some cases provide aesthetic qualities in food production. The inappropriate use of veterinary drugs can have adverse effects on animals, the environment, and human health.
The determination and efficient analysis of veterinary drugs is an important part of routine food quality control. The European Union (EU) and others have developed specific regulations to address these growing concerns. The requirements of low limits of quantification in diverse matrices, along with a wide variety of chemical classes and properties of veterinary drugs pose significant analytical challenges. Several methodologies exist which are typically limited in scope to specific chemical classes, labor intensive, and require extensive sample preparation and clean-up. This study presents a multi-residue, multi-class method describing a simple modified QuEChERs sample preparation procedure for the analysis of over 160 veterinary drugs using liquid chromatography-triple stage mass spectrometry (LC/MS/MS). Its initial performance was evaluated using three matrices: Bovine muscle, salmon fillet, and milk. The method is sensitive and robust, and is able to detect, confirm, and quantify the veterinary drugs below their required EU and US FDA maximum residue limits (MRLs).

P-44 Applications of the Q Exactive Focus Quadrupole-Orbitrap Mass Spectrometer at the Iowa State University College of Veterinary Medicine laboratory

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The high-resolution accurate-mass (HRAM) capabilities of the Thermo Q Exactive mass spectrometers make them extraordinarily versatile tools for a broad range of quantitative and qualitative analyses. At the Iowa State University College of Veterinary Medicine, we have successfully used this instrumentation to solve problems in a variety of application areas including: 1) targeted quantitation with UHPLC separation, 2) targeted quantitation with a PaperSpray source, 3) untargeted metabolomics, 4) targeted metabolomics using the Biocrates AbsoluteIDQ p400 kit, 5) targeted forensic screening of 300+ drugs in biofluid matrices, 6) general unknown screening in biofluids and tissue extracts, 7) structure elucidation of unknowns in drug seizure cases, 8) structure elucidation of unknowns in biological extracts and fractions, 9) tissue chemical imaging using desorption electrospray ionization (DESI), and 10) direct analysis of surfaces using a liquid micro-junction surface sampling probe (Flowprobe). Brief examples of each of these applications will be presented and the advantages of HRAM technology for each application area will be discussed.

P-45 Effectiveness of accelerated solvent extraction compared to QuEChERS methods for the multiresidue analysis of pesticides in organic honey by GC-MS/MS

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Honey is a natural product that is widely used for both nutritional and medicinal purposes. It is generally considered a natural and healthy product of animal origin, free of impurities. However, honey can be contaminated via pollen collection by bees and by direct treatment of hives with insecticides, fungicides, and acaricides. The European Union prevents the use of these pesticides in honey production, and has strong regulations for organic production to prevent contamination from other sources.

The determination of pesticide residues in honey is complicated by the presence of high concentrations of sugars and waxes, necessitating selective sample preparation techniques. Among the available extraction techniques, accelerated solvent extraction (ASE) offers shorter extraction times, reduced solvent consumption and, a higher rate of extraction. This study explores the effectiveness of ASE methods compared with QuEChERS, and finds that ASE methods can give superior LOQs and extraction linearity over the concentration range evaluated.

P-46 Ultra-Low Level Quantification of Pesticides in Baby Foods Using an Advanced Triple Quadrupole GC-MS/MS

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The maximum residue level (MRL) for the majority of pesticide-commodity combinations is set at the default level of 10 µg/kg. However, a small number of pesticides and their metabolites may allow infants and young children to exceed the acceptable daily intake values. In this study, the quantitative performance of the Thermo Scientific™ TSQ™ 9000 Triple Quadrupole GC-MS/MS system was assessed for the analysis of ~200 pesticides in baby food at very low concentrations (as low as 0.025 µg/kg). A complete evaluation of method performance included, sample preparation, overall method suitability measured from pesticides recoveries, selectivity, sensitivity, linearity and long term robustness.
The method performance was tested in accordance to the SANTE/10518/2017 guidance document. All detected compounds, at the three spiking levels in both matrices satisfied all SANTE requirements. More than 97% of the target pesticide residues had recoveries between 70 – 120% at the 1 µg/kg spiking level. Over 90% or the target compounds had an LOI (satisfying all SANTE requirements) below 0.5 µg/kg, and over 60% below 0.1µg/kg – 100 times lower than the

default MRL. Compound linearity was assessed by injecting matrix matched standards in the range of 0.025 to 250 µg/kg in duplicate for both carrot/potato and apple/pear/banana. Both sets of linearity data showed R2 > 0.990 and RF % RSDs of <20% for over 96% of component peaks indicating excellent linear response. Robustness displayed over ~400 consecutive injections of sample matrix (1 g/mL), with SANTE compliance at the default MRL throughout.

P-47 Accurate Quantitation of Pesticides and PCB's in Grape and Onion Extracts using High-Resolution GC-Orbitrap Mass Spectrometry

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When using high-resolution mass spectrometry, the default acquisition mode is untargeted (full scan) meaning that all the ions are acquired at the same time across a specified mass range, making it simple to manage and giving the analyst the flexibility to decide post-acquisition which pesticides and ions to measure. The objective of this study was to evaluate the quantitative performance of GC Orbitrap for the analysis of pesticides and PCB's in two sample matrices of varying complexity. The high resolution capability in combination with low limits of detection increases the scope of the analysis without the need for optimization of individual compound acquisition parameters. The first aim of the study was to establish the limit of detection (LOD) of the main quantifier ion for the 95 compounds in both the grape and onion samples. All of the compounds had a LOD ≤ 2 ng/mL with the exception of binapacryl, captafol and propargite (LOD = 5 ng/mL) in both grape and onion samples. Quantitative linearity in matrix was assessed across a concentration of 1-200 µg/Kg. In all cases, the coefficient of determination (R2) was >0.99 and the average response factor RSD% was < 20% for each analyte from its LOD to 200 µg/Kg in both the grape and onion matrices. To assess the accuracy of quantitation a grape and onion sample were analyzed blind after being post-spiked with compounds at concentrations varying from 0.5-100 µg/Kg.

P-48 Direct Determination of Cationic Polar Pesticides in Fruits and Vegetables using Ion Chromatography and MS/MS or High Resolution Accurate Mass Spectrometry

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Determinations of polar cationic pesticides, such as chlormequat, diquat, mepiquat, paraquat, morpholine, and trimethylsulfonium are challenging due to difficulty in using generic multi-residue methods. However, the Quick Polar Pesticides (QuPPe) method has been recently demonstrated on anionic pesticides determinations methods by IC-MS/MS.Here we demonstrate direct determinations of quaternary amine pesticides in homogenized fruit and vegetable samples using cation-exchange chromatography with serial detection by suppressed conductivity and mass spectrometry. These techniques are demonstrated by targeted MS/MS on a triple quadrupole mass spectrometer and by high resolution accurate mass spectrometry (HRAM MS) in full scan and MS/MS. wenty-three pesticides were determined in the targeted MS/MS method with a 20-min run time. The method was sensitive (triple digit ng/L to single digit µg/L), and robust (>100 injections of a prune sample). Fast determinations of 6 cationic pesticides were achieved with a 12 min run using HRAM mass spectrometry. Mepiquat, trimethylsulfonium, morpholine, and chlormequat exhibited good chromatographic resolution with Rs >2. The method was also developed on a prototype cation-exchange column optimized for the chromatographic separation of diquat and paraquat. The six pesticides had good accurate mass, meeting the SANTE requirements of <5 ppm. The homogenized food samples did not contain native cationic pesticides of interest. Sensitivities were measured in the single digit µg/L or less range by spiking pesticides to the samples. Good accuracy was found, with recoveries of spiked in reagents in the standards and the samples within 80 to 120%.

P-49 Rapid Determination of Ethynylestradiol (17aEE2) in Wastewater Using EQuan MAX Plus LC-MS System and Q Exactive Focus Hybrid Quadrupole – Orbitrap MS

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The occurrence and effects of endocrine disrupting compounds (EDCs) in aquatic environments is a significant concern. Of the many EDCs, 17αEE2 is recognized as possessing the greatest estogenic potency and risk to freshwater ecosystems and drinking water resources. Due to its environmental significance, 17αEE2 was incorporated into the EU Water Framework Directive, with a stipulated Limit of Detection of 35 pg/L, which presents a significant analytical challenge. Current methods involve large-volume SPE; normal phase SPE clean up and size exclusion fractionation, which take considerable time, expense, and sampling logistics. The aim of this work is to assess the possibility of using on-line solid phase extraction and a Thermo Scientific™ Q Exactive™ Focus hybrid quadrupole-Orbitrap™ mass spectrometer, for the determination of 17αEE2 at the WFD LoD. Seven replicate injections of the 100 pg/L standard were used for the calculation, with a t-test value of 3.143, giving an IDL of 19 pg/L. The method was calibrated between 100 and 2500 pg/L with a correlation coefficient of 0.9993. The method was applied to a sample of Scottish sewage effluent and 17αEE2 detected at 400 pg/L, which is in the range of values reported in the literature. Interestingly, the peak was immediately followed by a second peak of similar area and MS2 spectrum, showing identical diagnostic masses, and was tentatively identified as 17αEE2, which is significantly less estrogenic than 17αEE2. It can be inferred that other using methods that fail to chromatographically resolve the two isomers, would in this instance, report the 17αEE2 concentration with an approximate 100% positive bias, and have possible implications for regulatory compliance.

P-50 A Validated Analytical Method for Quantitative Determination of Pesticides and Mycotoxins in Cannabis by Liquid Chromatography Tandem Mass Spectrometry

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Subsequent to the legalization of use of cannabis for both medicinal and recreational purposes in certain states within the US, quantitative analysis of pesticide residues and mycotoxins in commercial cannabis is of great importance to scientific researchers, scientific instruments manufacturers, cannabis industry stakeholders and government regulatory institutions. Besides the complexity of the cannabis matrix, the more stringent regulatory action limits for both pesticides and mycotoxins from certain states, have furthered the analytical challenge required to accurately and reliably quantify these pesticides and mycotoxins. Nonetheless, the need for simple, robust, reliable and validated methods for pesticide analysis with good data reproducibility and accuracy are critical to all stakeholders in the cannabis industry. This work shows a validated and quantitative analytical method for analysis of 70 pesticides, including compounds typically analyzed by gas chromatography, and mycotoxins in cannabis flower using liquid chromatography coupled with tandem mass spectrometry. Limits of quantitation for all pesticides and mycotoxins were below all current regulatory action limits for pesticides and mycotoxins in cannabis in USA. Good reproducibility and precision were obtained for all analytes with %RSD ≤ 23 for all pesticides in the cannabis matrix without the use of an internal standard. Signal response stability studies carried out for over a 5-day period for 500 injections showed good reproducibility with a relative standard deviations (%) within 3% to 25% for all pesticides and mycotoxins using an internal standard.

P-51 Measurement of pesticides in apple juice using LC-MS/MS with fast and modified QuEChERS extraction method with volatile buffers

Avinash Dalmia

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Pesticide residue analysis is performed to ensure food safety by confirming that produce is in compliance with maximum residue limits. Liquid chromatography coupled to tandem mass spectrometry (LC-MS/MS) with QuEChERS is most widely used method for analysis of pesticide residues in food. Currently, QuEChERS uses non-volatile salts such as magnesium sulphate in combination with sodium chloride and other salts to induce salting out effect during pesticide extraction step. Unfortunately, these salts have low vapor pressures and tend to deposit in MS interface resulting in loss in sensitivity and higher maintenance. In this work, we report results for pesticide analysis in apple juice using a LC-MS/MS system with modified QuEChERS method with volatile buffers with excellent long term stability. The recovery for majority of pesticides in apple juice was in acceptable range of 70-120 % using the modified QuEChERS extraction method with

volatile buffers. The RSD of response for most of pesticides over 24 hr. was less than 5 % using injection of apple juice extracts obtained from modified QuEChERS method. Our results demonstrated that the combination of self-cleaning laminar flow interface in an electrospray source and modified QuEChERS extraction method would reduce maintenance needs of the LC-MS/MS based method for pesticide analysis in apple juice. The detection limits for pesticides ranged from 0.1 to 5ng/mL which meets the FDA mandated maximum residue limit of 10 ng/mL for pesticides in apple juice.

P-52 Rapid Determination of Cannabis Terpenes by GC/MS using Headspace Sample Introduction.

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Over 100 terpenes are known to contribute to the unique aroma of many different cannabis strains that range from fruity to woody and finally, skunky. The terpene profiles, including their relative abundances are useful in strain identification/validation but also useful in determining the therapeutic effect for medical purposes. It has been said that the terpene profile makes a significant contribution to both the recreational and medical effects of cannabis. For example, myrcene, the most abundant terpene found in cannabis has been reported to reduce inflammation and chronic pain, while the therapeutic properties of limonene are responsible for a calming effect.
Fifteen different terpenes are responsible for the aroma/therapeutic properties of the cannabis volatile fraction however; there is an increasing interest in the identification of the minor components as well. The increase in analysis time needed to completely separate the volatile fraction is a major stumbling block to its common use in the laboratory, since the lab is usually backlogged with samples and sample throughput is paramount in the analytical test lab. The focus of this study is to target up to 42 terpenes found in cannabis with attention to the speed of analysis and robustness, while maintaining excellent chromatographic separation and using mass spectrometry to assist with compound identification. Pressure Balanced headspace sampling was used due to its ease of sample preparation and accurate delivery of a precise aliquot to the GC/MS system for analysis. Data results such as chromatograms, spectra, calibration curves and repeatability will be shown.

P-53 Determination of Sugars in Animal Feed using HPLC-ELSD

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Animal feeds can contain sugars from a variety of sources such as plant material and binders such as molasses. Sugar content can vary widely depending on the type/maturity of grains used and the amount/type of molasses added. Even comparing a single type of grain, the sugar content can be greatly affected by growing and harvesting conditions. Because of this variability, sugar content can be difficult to predict, and testing is often required to ensure nutritional and dietary requirements are met. In this work, a HPLC-ELSD method was evaluated for the analysis of six free nutritional sugars in animal feed: fructose, galactose, glucose, sucrose, maltose, and lactose. Assay performance including precision and recovery were evaluated and met the acceptance criteria. A Luna® Omega SUGAR LC Column was selected for its ability to retain polar sugar molecules using HILIC conditions and differentiate individual sugars. The HPLC methodology allowed for a simple sample extraction with ethanol and water followed by filtration. In contrast with RI detection, ELSD enabled use of a gradient chromatographic method, producing sharper peaks and baseline resolution of glucose and galactose.

P-54 Determination of Aflatoxin B1, B2, G1 and G2 in Grain using Solid Phase Extraction and LC-MS/MS

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Produced by various fungi, Mycotoxins are very harmful toxins found regularly in feed and grain products. Mycotoxins, result in both acute and chronic health effects in humans and livestock when consumed. With the globalization of the food supply, Mycotoxins from grain is a global concern for both developing and developed countries. In this study, we present a rapid and sensitive method for mycotoxins per the new China GB method GB2009.22-2016 that can be used

on both HPLC and UHPLC platforms. Specifically, Aflatoxin B1, B2, G1 and G2 in grain are extracted using Strata®-X solid phase extraction (SPE) followed by a rapid LC-MS/MS method using a Kinetex® 1.7 µm C18 Core-shell LC column. The SPE sample cleanup successfully removes interferences from the grain resulting in great recoveries meeting the assay acceptance criteria. Additionally, the high efficiency and selectivity of the core shell column produced excellent baseline separation of the 4 Aflatoxins for more accurate quantitation.

P-55 Analysis of Polyether Antibiotics in Animal Feeds by HPLC with Post-Column Derivatization

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Polyether Antibiotics are commonly used for preventing coccidiosis and other infections in poultry and for improving feed efficiency for beef cattle and swine. The use of Polyether Antibiotics is strictly regulated, with only specific ionophores approved for use in feeds intended for different animals.
Analysis of Polyether Antibiotics by HPLC with post-column derivatization and UV/Vis detection has been proven to successfully identify and quantify Monensin, Narasin and Salinomycin in medicated feeds, supplements and premixes as well as to determine trace contamination levels in non-medicated feeds [1, 2].
Post-column derivatization of Polyether Antibiotics is done using highly acidic Vanillin or DMAB reagents. The Pinnacle PCX derivatization system (Pickering Laboratories, Inc.) has an inert flow path and automated system wash capabilities that make it uniquely suitable for handling corrosive reagents. The two-pump system is recommended to extend reagent stability, but the single-pump system for this application is also available.
Adding a Fluorescence detector to the instrumentation allows for using the same extraction procedure and HPLC conditions to also determine Lasalocid, which doesn't require post-column derivatization.

P-56 Evaluation of an Alternative Ion-Pairing Chromatography Technique for the LC-MS/MS Analysis of Underivatized Biogenic Amines in Ground Beef

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In a recent development in ion-pairing chromatography, it was shown that addition of the ion-pairing agent into the sample instead of the mobile phase can produce satisfactory retention of highly hydrophilic compounds on a reversed-phase column. This technique was employed for the analysis of eight biogenic amines (2-phenylethylamine, cadaverine, histamine, putrescine, spermine, spermidine, tryptamine and tyramine) on a Kinetex® C18, 100 x 2.1 mm, 5 µm column. Samples were prepared with a mixture of n-octane-1-sulfonate and 0.1% aqueous formic acid. The mobile phase consisted of 0.1% formic acid in water and 0.1% formic acid in methanol. A shallow gradient resolved all compounds within 7.5 mins. The sample preparation involved solid-liquid extraction of ground beef with dilute trichloroacetic acid. After the extraction, the supernatant was separated and subjected to an SPE procedure with weak cation exchange polymeric SPE media. Various extracts were fortified with biogenic amines to contain different concentrations, ranging from 100 to 1000 ppb. The data collection was carried out on a SCIEX 4000 QTRAP® system equipped with ESI source operating in positive polarity MRM mode. The peak area from various analytes were compared to determine a simple LOD based on signal-to noise ratio. Furthermore, the analytes' peaks were closely scrutinized for retention time shifts and peak shape abnormalities such as tailing, splitting and/or fronting. The ion-suppression study identified two sections of the chromatogram which produced the highest levels of signal suppression. These areas are outside the analytes' elution window and were diverted to waste.

P-57 Analytical method development and validation for the analysis of pesticides in cereals and oilseeds using QuEChERS cleanup

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Effective sample preparation procedures are necessary to reduce interferences as there can frequently be significant background peaks in chromatograms collected by less selective techniques such as GC/ECD and even GC single quad MS. QuEChERS has become a popular technique for sample preparation that continues to be used for new applications.

A new method using QuEChERS sample cleanup for the analysis of 41 pesticides in cereals and oilseeds by GC/MS or GC/ECD analysis is described. Assessments of the importance of wetting samples, and the effects of including C_{18} in QuEChERS sample preparation on extraction efficiency, and reproducibility measured by RSD, and extent of background peaks in GC single quad and GC/ECD chromatographic data were performed. Reducing sample size from 30 to 7.5 g resulted in statistically significant improvement in extraction efficiencies for a majority of compounds. Wetting samples and increasing the period of QuEChERS exposure during shaking significantly reduced background, thereby improving the overall sensitivity for metolachlor, chlorpyrifos, flufenacet, cyprodinil, and pendimethalin. Final extraction efficiencies corrected for instrument variability were found to be 45 to 65 %, with RSDs below 15 % for 39 of 41 compounds.

P-58 Quantitative Analysis of Erythromycin and Tylosin in Honey Utilizing RapidFire Mass Spectrometry

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New advances in technology are accommodating the need for higher sample throughput while maintaining or improving detection levels. Mass spectrometry is predominantly the analytical tool of choice for most of the U.S. Food and Drug Administration's (FDA) regulatory field laboratories. Traditionally, most mass spectrometers were coupled with a device that introduced the sample (i.e. high performance liquid chromatography, gas chromatography, etc.) into the mass spectrometer. Although effective, these devices necessitate a considerable amount of time to provide acceptable results. Additionally, tedious and labor intensive sample preparation and extractions are often required for analysis using the technologies. In efforts to try and increase sample capacity, and further enhance the public's safety with respect to food safety, the FDA's Arkansas Laboratory has focused its efforts on developing a rapid quantitative screening method for tylosin and erythromycin in honey using mass spectrometry. Our research has demonstrated that an automated solid phase extraction system that is directly coupled to a mass spectrometer is an effective alternative to the traditional liquid-liquid extractions and LC/MS/MS analysis. This screening method can perform unattended and automated sample extraction and quantitative analysis of these drug residues in honey on a triple quadrupole mass spectrometer. Furthermore, each individual sample can be extracted and analyzed in less than 15 seconds which will enable laboratories to accommodate a larger volume of samples and shorten sample turnaround times.

P-59 Pesticide residue analysis in Hops: Four different procedures of sample preparation are currently used. Is one of them more useful than others?

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The hop plant belongs to crops with intensive chemical protection against wide scale of bacterial diseases, fungus and mildew, virus diseases, as well as pests and parasitic invasion. Nowadays, four different sample preparation procedures are commonly used for pesticide residues analysis in hops. These methods are based on completely different approach for sample preparation using various solvents for extraction pesticide residues from matrix and the clean-up step is also unique for each method. One of those is recently published QuEChERS method in combination with specially optimized mixture for dispersive solid phase extraction. The AHA collaborative trial showed that all methods give more or less comparable results. The critical evaluation of each method is desirable for the purpose to choose method which is most effective and easy to handle.
In our initial set of experiments we focused on comparison of all published methods. Our attention was focused on the ability of each method to effectively minimize co-extraction of matrix components (chlorophyll, resins, bitter acids) which can mostly cause massive signal suppression, elevated background, and other negative matrix effects. The recoveries of fifty five spiked pesticides were also evaluated for each method. The final evaluation involved, apart of these values, parameters such as time demand, total solvent consumption and ability to analyze also some trouble making pesticides (e.g. flonicamid metabolites TFNA and TFNG).

P-60 Investigation into the concentrations and sources of nitrates and nitrites in US milk powders

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Milk powders in the United States may contain nitrates and nitrites from several potential sources. These sources include the ingestion of nitrates and nitrites of dairy cows during grazing and drinking, nitric acid used during the sanitization of dairy equipment, and the production of nitrous oxides in directly heated spray dryers. Recently milk powders manufactured in the United States have been rejected from import to other countries due to having nitrite concentrations greater than 2 ppm. To date, the concentrations of nitrates and nitrites in milk powders in the US is unknown. In order to investigate the nitrate and nitrite concentrations present in US products, 83 milk powders were collected from local and online retailers from 2015-2018 and 71 commercial milk powders were obtained from blinded production facilities in the US and 3 other countries. The commercial samples also contained milk powders spray dried at three different temperatures (low, medium, high). Nitrate and nitrite concentrations were determined using ion chromatography with conductivity and UV detection. Carbon and nitrogen bulk isotope analyses of the milk powders were performed using Isotope Ratio Mass Spectrometry (IRMS) to gain further information about the diet (wheat/maize) and fertilization (organic/synthetic) of the feed crops of the dairy cows. Additionally, nitrate and nitrite salts purified from the milk powder extracts were also analyzed using IRMS. The combination of nitrate/nitrite data and the results of the stable isotope analysis provides valuable information on the sources of nitrate/nitrite in the milk supply.

P-61 Protecting your pet from euthanasia drug: Low-level quantitation of pentobarbital in pet food and pet food ingredients by UPLC-MS/MS

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Recently, Pentobarbital, a euthanasia drug was found in certain dog food, which claimed the life of one dog and made other dogs ill. This prompted into certain pet food recall and FDA alert to pet owners about possible contamination in pet food. Pentobarbital contamination in pet food raised the need for accurate and low level contamination of pentobarbital in pet food and pet food ingredients. Traditionally, pentobarbital was analyzed by GC-MS/MS involving derivatization step. However, due to concerns associated with uncertainty during derivatization step, LC-MS/MS was chosen for analysis. In the current method, pentobarbital was extracted from dry pet food and pet food ingredients using methanol. In the next step, extracts were cleaned up by SPE cartridges. Standard solution calibrations were prepared for the analysis in order to expand the applicability of method to different matrices. Chromatographic separation was carried out on reversed phase column. The samples were analyzed by UPLC-MS/MS equipped with electrospray ionization on QQQ mass spectrometer. Two MRM transition were selected for the analyte. The linearity, method detection limits and method quantitation limit were studied. Method validations were performed in dry pet food, porcine liver and animal fat. Based on the validation data, accurate quantification of pentobarbital below 1 ppb was reported in all matrices.

P-62 Determination of Polycyclic Aromatic Hydrocarbons in a Yerba Mate Standard Reference Material by GC-MS

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Ensuring food safety requires routine screening of foodstuffs for potentially hazardous contaminants. Matrix relevant quality control materials, such as certified reference materials, are necessary to develop and validate methods and/or confirm results for the detection of contaminants. Contamination from carcinogens such as polycyclic aromatic hydrocarbons (PAHs) may be of concern for foods prepared at high temperatures. For example, the leaves of yerba mate are often dried over wood burning fires. The dried leaves are then steeped to prepare a hot drink which may contain high concentrations of PAHs. The National Institute of Standards and Technology (NIST) has recently released Standard Reference Material (SRM) 3253 Yerba Mate Leaves to address the need of measuring PAHs in food matrices. SRM 3253 was prepared from commercially available yerba mate that was ground in an ultracentrifugation mill, packaged, and

irradiated to prevent microbial growth. Soxhlet extraction and pressurized fluid extraction were used to isolate the PAHs from the yerba mate, and solid phase extraction was used for sample cleanup prior to GC-MS analysis. SRM 3253 was found to be homogenous with respect to the concentrations of the PAHs using stratified random sampling across the entire batch of material. Assignment of certified and reference values was based on combining results from two or more independent methods and is reported on a dry-mass basis. SRM 3253 can be used as a quality control material for the determination of PAHs in botanical and food matrices.

P-63 Liquid Chromatography -Tandem Mass Spectrometry vs. Ion Chromatography -Tandem Mass Spectrometry for Glyphosate Residues Analysis in Botanical Matrices

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Analysis of polar pesticides such as glyphosate and its metabolites is very problematic due to the physiochemical properties of the molecules. The retention of anionic pesticides using reverse phase chromatography is difficult without derivatization and is not robust enough to implement in a QC laboratory. Although many methods utilizing different instruments and chromatographic approaches are now available, their robustness and performance when analyzing glyphosate residues in difficult matrices is questionable.
Two approaches to analyze polar pesticides in dietary botanicals were assessed. LC-MS/MS method utilizing polyvinyl alcohol with quaternary ammonium groups column was evaluated against an IC-MS/MS method separating the analytes on a polymer based column. Glyphosate, AMPA, MPPA and glufosinate were either extracted from botanical matrices according to the QuPPe method (LC-MS/MS approach) or extracted in water/cold methanol (IC-MS/MS method). The analytes were identified and quantitated by Thermo Altis mass spectrometer with the use of isotope-labeled internal standards.
Both methods delivered satisfactory results, although the IC-MS/MS approach showed better robustness when analyzing complex botanical matrices. Established limits of detection and quantitation using both methods for glyphosate in the matrix were below regulatory limits in the EU.

P-64 Estimation of Persistent Organic Pollutants in Lakes of Broknes Peninsula in East Antarctica

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The present studies was designed to evaluate the concentration of persistent organic pollutants (POPs) in lake water samples, collected from five selected locations on Broknes peninsula during austral summer in 34th Indian Scientific Expedition to Antarctica (ISEA) from 2015-2016. A total of 15 lake water samples were collected. In these samples, thirty four compounds of pesticides and thirty compounds of polycyclic aromatic hydrocarbon (PAH) & polychlorinated biphenyls (PCBs) were estimated. Lake water samples were processed using a liquid-liquid method solvent extraction, cleaned-up and concentrated. The concentrated sample then injected in to GC-MS/MS along with standards and then quantitative analysis was performed. Pesticides residue levels found in lake water samples varied from 10.33 pg/ml to 70.00 pg/ml and PCBs & PAHs levels from 10.33 pg/ml to 50 pg/ml.
After evaluating the large number of compounds of POPs in lake water, it has been observed that the Broknes peninsula in east Antarctica has trace amount of POPs which is an alarming situation and needs to be investigated further to maintained pristine environment in Antarctica.

P-65 Multiclass Veterinary Drugs Analysis in Swine Muscle and Hen Eggs Using Ultivo Triple Quadrupole LC/MS System

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Veterinary drugs have been widely used to treat or prevent diseases and enhance growth and feed efficiency. There have been world-wide concerns that veterinary drug residues in the products at the time of consumption can cause adverse

effects to humans. Government agencies have set limits for allowable veterinary drugs. Due to the complexity of the matrices and characteristics of different classes of veterinary drugs, it is challenging to detect multiclass of analytes in one run. A typical standard method covers one class or a small cohort of similar classes of veterinary drugs. To efficiently monitor veterinary drugs, the ability to detect multiclass of analytes is highly desired. 151 veterinary drugs were analyzed in swine muscle and hen egg matrices and cleaned using EMR-Lipid for efficient fatty components removal. The 9-level calibration was run from 0.1 ng/g to 40 ng/g. The method benefits from the fast polarity switching in a dynamic multiple reaction monitoring (MRM) fashion. The analytes were separated using a, 3.0 x 150mm, 1.8mm, C-18 column, with water and acetonitrile as mobile phases, both modified with 0.2% formic acid. Analysis was performed using Ultivo triple quadrupole LC/MS system. Most of the linear calibration curves had R^2 > 0.99, with 1/x weighting. ~87% of analytes could be accurately quantified at 1 ng/g with S/N >10 for both quantifier and qualifier. The precision was excellent. ~92% of the compounds had %RSD < 20% at 5 ng/g (n=7). The new, robust Ultivo provided outstanding method repeatability over multiple days of sample analysis.

P-66 Quantitative Determination of Multi-Class Multi-Residue Pesticides in Edible Oil Using Captiva EMR-Lipid Cleanup by GC/MS/MS

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Multi-class multi-residue pesticides analysis in oily sample matrix is always quite challenging, especially for more hydrophobic compounds, due to the difficulty of pesticides extraction from high fatty matrices and the risk of analyte loss during the matrix cleaning. Captiva Enhanced Matrix Removal – Lipid (EMR-Lipid) cartridges are implemented in a solid phase extraction (SPE) format providing pass-through cleanup for highly selective lipid removal without impacting analyte recovery. The EMR-Lipid sorbent specifically interacts with the unbranched hydrocarbon chains of lipid compounds using a combined mechanism of size exclusion and hydrophobic interaction. In order to extract pesticides from oil, an Acetronitrile based solvent extraction was used with a 30 minutes of vigorously mixing. After extraction, the ACN supernatant was mixed with 20% water, and the mixture was loaded onto Captiva EMR-Lipid cartridge for pass-through cleanup. Through this cleanup, the fatty sample matrix can be cleaned efficiently with >90% of matrix residue being removed. Due to the highly selective lipids removal by EMR-Lipid sorbent, the target pesticides were not negatively impacted by the matrix cleaning. Excellent recoveries (70-120%) and precision (RSD<20%) were achieved for the majority of analytes. Additionally, the samples with EMR-Lipid cleanup also reduced sample matrix impact on GC/MS/MS analysis and improved instrument analysis reproducibility for labile pesticides. Cleaner samples also protected GC/MS/MS flow path from fast deteriorating with multiple injections, and thus reduced the system maintenance frequency and instrument downtime.

P-67 Approaches to Analyzing for Pesticide Residues in Cannabis by GC/Q-TOF and GC/QQQ

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Previously, we reported on the screening for pesticide residues in confiscated cannabis samples using a high resolution accurate mass GC/Q-TOF instrument. The method used a “Find by Fragments” algorithm in combination with a Pesticide Personal Compound Database and Library (PCDL). The software could screen for more than 850 pesticides in the PCDL by extracting out characteristic accurate mass ions for each compound at its locked retention time. In sixteen different samples, an average of almost six pesticides and related contaminants were found by this method. More recently, we have developed a quantitative method for 85 pesticides, most of which are halogenated. A major problem with analyzing pesticide residues in cannabis is that extracts contain very high levels of terpenes and cannabinoids which cannot be removed without losing pesticide residues as well. The most successful extraction technique involves a 500-fold dilution of the extract. Of course, this much dilution requires an extremely sensitive mass spectrometer to detect the target compounds. This is possible using a GC/QQQ and GC/Q-TOF each of which has a high efficiency source that makes the instrument significantly more sensitive than previous models. The GC/QQQ works well for this analysis, even with the 500-fold dilution. The next step is to see if this dilution approach will work with the new GC/Q-TOF with its improved sensitivity and much higher resolution.

P-68 GC/Q-TOF Workflows for Comprehensive Pesticide Analysis

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This presentation demonstrates three complementary GC/Q-TOF workflows for the comprehensive analysis of pesticides and related contaminants in environmental samples: 1) Target quantification, 2) Suspect screening using high resolution accurate mass GC/Q-TOF data, and 3) Non-target screening employing spectral deconvolution and library searching. A GC/Q-TOF was used to analyze 51 water samples taken from the Sacramento/San Joaquin River delta in California before, during and after two rain events. After filtering, contaminants in the water extracts were isolated by solid phase extraction and concentrated by solvent evaporation. The filters were extracted to recover contaminants bound to particulates. All extracts were analyzed by GC/Q-TOF in the NCI and EI modes. NCI was used for quantitative analysis employing a validated method for 21 pesticides. Sixteen of the target pesticides were found in the extracts. The EI results were analyzed using a Find by Fragments (FBF) workflow which screened for several hundred pesticides and other contaminants contained in Agilent's Pesticides and Environmental Pollutants Personal Compound Database and Library (PCDL). Forty-one additional suspects were identified using this technique, with most being confirmed by the analysis of standards. Non-target screening employed the Agilent MassHunter Unknowns Analysis Software. This software first deconvolutes the spectra in the chromatogram and searches the deconvoluted components against a mass spectral library. Five pesticides and one transformation product (TP) not found by the first two GC-Q/TOF approaches were tentatively identified in Unknowns Analysis. In addition, several halogenated and non-halogenated organophosphorus flame retardants, several phenolic antioxidants, and various organohalogen compounds were tentatively identified.

P-69 Screening for 1000 Pesticides and Environmental Contaminants using a new High Resolution Accurate Mass GC/Q-TOF

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It is necessary to measure pesticides and environmental contaminants in our food and environment to ensure human and wildlife safety. In the recent past this was done by GC coupled to a single quadrupole MS. In the last ten years, triple quadrupole MS has largely supplanted single quadrupole instruments in the lab because of their much higher selectivity. More recently, GC has been coupled to high resolution accurate mass (HRAM) MS taking advantage of its high selectivity, full spectral acquisition and ability to measure an unlimited number of analytes. This presentation discusses the use of a new HRAM GC/Q-TOF MS with a new personal compound database and library (PCDL) which contains exact mass spectra for nearly all GC-amenable pesticides and many important environmental contaminants (~1000 compounds). Three workflows are used: 1) target compound analysis with quantification, 2) suspect screening for all compounds in the PCDL and 3) nontarget screening using deconvolution and library searching. This approach has been applied to surface waters, marijuana and food extracts. A novel high efficiency source design offers much better sensitivity than previous instruments which makes it possible to dilute an extremely dirty sample such as a marijuana extract and still measure contaminants at relevant levels. This helps to maintain inlet, column and source cleanliness.

P-70 Multiresidue Pesticide Analysis with the Agilent Intuvo 9000 GC and Agilent 7000D Triple Quadrupole GC/MS

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As pesticide use has increased so has the level of concern among environmentalists, regulators, and consumers. Regulations regarding the maximum limit of pesticide residues that can be found in or on food (MRLs) have been established nearly worldwide, including countries in North America (United States and Canada), Europe (European Union), Asia (Japan), and Australia. In the United States, MRLs can range from 0.02ppm to 100ppm depending on the matrix and pesticide in question while the European Commission has a default value of 0.01ppm.

The analysis of pesticides in food can quickly become complex with increasing target compound and commodity lists and decreasing detection limit requirements. Having a robust method on an easy-to-use platform that integrates seamlessly to a large database is desired to facilitate this analysis. Coupling Agilent's 7000D Triple Quadrupole GC/MS to Agilent's newest GC platform (Intuvo 9000 Gas Chromatograph) delivers a streamlined workflow that involves the implementation an inert microfluidic retention gap (guard chip) for multiresidue pesticide analyses.
Calibration curves for 25 pesticides in varied matrices showed excellent linearity for concentrations ranging from 1 ng/mL to 500 ng/mL. Excellent response and peak shape consistency was obtained with the implementation of the Intuvo guard chip which protects downstream components and eliminates the need to trim the column after matrix evaluation. The average accuracy reported for this standard was 80% with an RSD of less than 10%. With regular maintenance, including liner and Intuvo guard chip replacements, peak shape, response, and analyte calibration can be preserved for over 500 injections.

P-71 Direct Analysis of Glyphoaste and its Metabolites, in Search of a Robust LC-MS/MS Method for Food and the Environment

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Glyphosate is the active ingredient in the popular herbicide roundup and is used throughout the world. Recently, its safe use has come into question. This has heightened the demand for a sensitive method at the low ppb level for food and even lower levels for environmental water analysis. LC/MS/MS using triple quadrupole technology can provide both the sensitivity and selectivity needed. In this presentation, we examine the conditions required for a robust analysis and this prolific herbicide and its metabolites AMPA, HEPA, MPPA, glufosinate and its metabolite n-acetyl glufosinate. The direct ion exchange separation of both a quaternary amine bound to polyvinyl alcohol column and a quaternary amine bound to polystyrene divinylbenzene will be described and compared. In addition, the deactivation of the system for reproducible from laboratory to library will be discussed. Finally, a robust method must have a good sample preparation procedure that does not require extensive processing. Methods for different foods and drinking water will be presented.

P-72 Using Modified QuEChERS for the Extraction of Pesticide Residues in Botanical Ingredients Using GC-MS/MS

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Botanical ingredients have been used for centuries in Asia and their increased use in the US is a multi-billion-dollar industry. Many of these supplements have been concentrated and dried from original plant matter that was treated with pesticides. Historically, the amount of consumption has been very low, but as these ingredients are added to an increasing number of products the overall potential exposure to pesticides requires further investigation. Validated methods are needed to determine pesticide residue levels in these finished products. This study is based on work done by Douglas G. Hayward and Jon W. Wong who demonstrated the feasibility of extracting and identifying pesticides in different dried plant products (Hayward *et al*., 2015). The preparation uses a Quick Easy Cheap Effective Rugged Safe (QuEChERS) extraction procedure in addition to solid phase extraction (SPE). 10 milliliters of water and 10 milliliters of acetonitrile are added to one gram of the dried, finely powdered supplement, along with 4 grams of anhydrous magnesium sulfate and 1 gram of sodium chloride. The different phase layers are expressed using a centrifuge and 1.25 milliliters is removed and extracted through a solid-phase extraction (SPE) column containing both graphitized carbon black and a bottom layer of primary-secondary amine with anhydrous sodium sulfate to remove any residual water. Gas chromatography is well suited for the analysis of these samples with the ability to resolve multi-components which include coextracted interferences. The samples chosen; ginseng, green tea, chamomile, represent a complex matrix which requires coupled mass spectrometry in the determination of trace level pesticides. This work reviews the extraction and analysis of 165 pesticides to include multi-matched calibration standards, calibration curves and mean pesticide recoveries for each matrix.

P-73 The LC-UV Analysis of 16 Cannabinoids of Interest in Commercially Available CBD Oils

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More than 100 cannabinoids have been isolated from cannabis in addition to the five most commonly tested: THC, THCA, CBD, CBDA, and CBN. While methods have been published that show the separation of these major cannabinoids, many do not take into account the possibility of interference from other cannabinoids that may be present. This is most problematic in concentrates where minor cannabinoids can be enriched to detectable levels that were not observed in the flower. Additionally, some terpenes have been shown to absorb UV light at 228 nm, the wavelength cannabinoids are typically detected, which can result in an additional source of interference. In this study, the LC-UV separation of 16 cannabinoids of interest was performed while monitoring for the potential impact from minor cannabinoids and terpenes on reported potency values. The method is applied to commercially available CBD oils that have recently become suspect due to inaccurate label claims.

P-74 A Perfect Pairing: HILIC and Superficially Porous Particles in Food Analyses

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In food analyses, separations with good selectivity accompanied by highly sensitive detection are desired. This can be difficult to achieve with polar compounds analyzed by reversed-phase liquid chromatography (RPLC). Hydrophilic Interaction Liquid Chromatography (HILIC) encompasses a wide range of stationary phases that provide suitable retention and selectivity for many classes of polar molecules. HILIC typically provides greater MS sensitivity because highly organic mobile phases are used. When approaching RPLC, there are limited retention mechanisms to consider. Aliphatic hydrocarbon stationary phases and phenyl-type stationary phases are well-understood. Aliphatic hydrocarbon stationary phases (e.g. C18) provide van der Waals interactions between the phase and the analytes, while phenyl-type stationary phases (e.g. Biphenyl) provide primarily π-π interactions. HILIC phases encompass a wider variety of chemical interactions that are used with a high organic (typically buffered) mobile phase consisting primarily of acetonitrile and water. The intermolecular interactions between analytes and stationary phase can include, but are not limited to, hydrogen bonding, strong/weak cation or anion exchange, zwitterionic phases, and many proprietary phases that contain “polar embedded” groups. As a newer emerging technique, HILIC may seem initially challenging to implement. The objective of this poster is to deconvolute the phase chemistry of several HILIC stationary phases and set some basic ground rules for method development. We will also offer solutions for some common issues that are encountered with the use of HILIC, as well as show some examples of how to improve your HILIC separations.

P-75 Analysis of Bisphenols and Perfluorinated Compounds by LC-MS/MS Using Raptor 1.8 µm Columns

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There are myriad contaminants that have impact on our food and environment. In this poster we present separations performed by the Restek Raptor 1.8 µm column family on two classes of contaminants: bisphenols and perfluorinated alkyl acids (PFAs). Bisphenol A (BPA), widely used in the production of polycarbonate plastic and epoxy resins, is an endocrine disruptor that imitates naturally occurring hormones or acts as an antagonist which leads to harmful health effects. As public awareness increases, many products are advertised as “BPA-free,” but instead use BPA analogues that have similar physicochemical properties, which also have harmful toxicological profiles. An analytical method using a Raptor Biphenyl 1.8 µm column has been developed that can resolve BPA and its most prevalent analogues for monitoring human exposure to bisphenols. Similarly, PFAs are widely used across a broad range of products, such as firefighting foams, coating additives, textiles, and cleaning products. PFAs are resistant to degradation and have become persistent, ubiquitous environmental pollutants in soil, air, groundwater, municipal refuse, and landfill leachates. The potential adverse effect on animal and human health has been reported for long-chain PFAs, in particular, perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS). To phase out the application of long-chain PFAs, a wide range of short-chain PFAs are used as replacements, leading to rising levels of their disposition in environmental and human matrices. A Raptor C18 1.8 µm column is well suited to baseline separating many of the most common PFA residues.

P-76 Strategies for Automated Calibration Development and Quality Control Review

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Instrument calibration, required for any accurate quantitative calculation, is a trivial process when performed correctly, but is also full of easily overlooked stumbling blocks. To minimize the risk of error associated with improper calibrations, national and international guidance dictates a minimum number of calibrators and the threshold at which a measurement becomes an outlier. Evidence from individual laboratory practice, which may differ from some aspects of regulatory guidance, suggests that what some groups may be doing is alternatively described as "remapping" their detector with each batch of samples. This can be observed as calibration adjustments which may be made to improve apparent calibration quality, as specific figures of merit (e.g. R2; calibration standard deviations when back-calculated against the calibration; etc.). These types of calibration adjustments may also be made to adjust measured quality control sample results.
We present a *post facto* explanation for the calibrator minimum and provide recommendations for curve building, which include improved outlier detection for high-volume mass spectrometry laboratories. These data show both a theoretical/exemplar perspective, as well as practical considerations and results from production laboratories. Building on these data, and working across collections of batches over time, a set of real-time and retrospective data analytics and dashboards are also presented, as a mechanism to holistically assess the historical, current, and future (projected) quality state of the laboratory, and as an indicator for impacts in released results.

P-77 Comprehensive Cannabis Analysis Including Strategies for Extended Range Potency Measurement

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Cannabis for medical and adult use in Canada and the USA substantiates the need for robust and reproducible methods for analysis of Cannabis products for consumer health and safety. The analysis of Cannabis products has demonstrated that these matrices are some of the most complex and difficult to analyze, demonstrating common issues with interferences and ion suppressors. Analysis using both for over 60 pesticides residues will be discussed. A simple acetonitrile solvent extraction allows for the analysis of not only pesticides, but for additional Cannabis residues and active components including cannabinoids, mycotoxins and terpenes. Limits of quantitation (LOQs) for both solvent and Cannabis flower matrices have been established for compounds on several regionally regulated pesticide lists. Due to the exclusion of complex clean-up steps, the same extracts used for pesticide analysis can also be used to analyze for mycotoxins and the endogenous Cannabis cannabinoids and terpenes. Advanced data processing strategies are additionally described which allow potency assessment for a wide range of relevant cannabinoid profiles. This extended range potency strategy is demonstrated effective for quantifying cannabinoids from very low levels in product (<1% weight) to very high levels in product (>90% by weight). This novel approach serves the testing lab by allowing for a single extraction and injection step, and controlling the quantitation at the processing step, reducing the need for re-injections. This results in a comprehensive Cannabis solution to measure contaminants, potency and flavor profiles using a single instrument platform and a single sample preparation strategy.

P-78 Combining Non-Targeted SWATH® Acquisition with MRMHR for the Analysis of Veterinary Drugs in Tissue

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Veterinary drugs are commonly used in livestock breeding to prevent or treat infections of the animals and to ensure their optimal growth. Legal regulations define waiting periods between the application of active pharmaceutical ingredients and the release of the animals for food manufacturing. Veterinary drugs which still find their way into human nutrition represent a potential risk to human health. Abuse of antibiotics in animals may also contribute to the development of antimicrobial resistance.

Often, region-specific regulatory guidelines require accurate and sensitive screening and quantitation of veterinary drug residues in animal products. This work explores a versatile and sensitive workflow on the SCIEX X500R QTOF system which combines a nontargeted screening workflow using SWATH® data acquisition looped with highly selective MRMHR acquisition. Confident identification of veterinary drug residues according to European requirements is achieved by accurate precursor and fragment mass measurement and their compound specific ion ratios, as reported in the SCIEX OS software. A highly flexible, selective and sensitive LC-MS/MS method for the analysis of veterinary drugs in liver extract

is presented, using the SCIEX X500R QTOF high resolution mass spectrometer together with the SCIEX OS software for a combined non-targeted and targeted screening workflow.

P-79 QTOF for Compound Quantitation and Identification: Workflows Utilizing MRMHR and SWATH®

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In addition to robust routine quantitation, testing laboratories are increasingly tasked with confirmation of positive detections of pesticides or other residues. Application of LC-MS/MS with multiple reaction monitoring (MRM) has long represented the principal workflow for such analyses due to the high degree of sensitivity and selectivity imparted by the monitoring of unique MRM transitions. The work presented explores the additional advantages gained when leveraging High Resolution Accurate Mass (HRAM) mass spectrometric technology for both targeted quantitation as well as screening. The key advantages of this HRAM approach are realized in the streamlined MRM^{HR} workflow which achieves sensitive and selective quantitative data collection and processing for a targeted panel with concurrent collection and matching of MSMS spectra in the same injection. High confidence in compound identification is achieved through multiple points of matching including accurate precursor ion mass, isotope pattern matching, accurate fragment mass, ion ratio, chromatographic retention time, and library matching. The additional capability of SWATH Data Independent Acquisition furthers the ability to do screening workflows including searching MSMS and elucidating formulae and candidate structures. SWATH collects MSMS spectra from all peaks in a sample, so even low- level residues in complex matrices can be explored. Combined with the SCIEX All-in-One MSMS library with NIST, compound library coverage is expanded to allow for further probing of nontargeted peaks and corresponding MSMS spectra.

P-80 Examination of Waters from Various Points During Conventional and Advanced Treatment with Nontargeted Screening using High Resolution LC-QTOF-MS/MS

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Wastewater samples represent one of the most complex possible mixtures of anthropogenic and environmental organic contaminants, constituents, transformation products, and dissolved solutes. A workflow based on liquid chromatography coupled to a quadrupole-time-of-flight mass spectrometer (LC-QTOF-MS) was developed and applied to detect and identify suspect and unknown species in raw and treated water samples collected from a treatment plant. Candidate structure assignments were made based on experimentally derived high-resolution accurate mass data and MS/MS spectral interpretation (including comparison to spectral databases and in silico fragmentation predictions). Data were collected using a novel combination of SWATH (a Data Independent Acquisition scan) and IDA (Information Dependent Acquisition) scan types in a single acquisition. Corrosion inhibitors, artificial sweeteners, and pharmaceuticals were among the most concentrated components to be detected and identified in the samples. Distinct differences in contaminants detected were observed between raw, treated, and advanced treated waters. High resolution-accurate mass (HRAM) data combined with powerful software tools for nontargeted screening workflows are critical to achieve data which are able to inform streamlined, confident candidate species and structure identification.

P-81 Simultaneous Analysis of 25 Mycotoxins in Grain by LC-MS/MS

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Traditionally, different classes of mycotoxins required different sample preparation techniques, making the process laborious and time consuming. Presented is an integrated workflow to analyze 25 compounds simultaneously in one sample. This fast, robust, and reliable method was developed and validated in grain matrix as an important product for mycotoxin analysis. A fast purification method was used to cover the 25 kinds of mycotoxins. High resolution LC using a small particle size column was combined with high sensitivity detection using a SCIEX QTRAP® 4500 LC-MS/MS system. Multiple Reaction Monitoring (MRM) was used because of its high selectivity and sensitivity. The Scheduled MRM™

algorithm used automatically optimized dwell times and cycle times for best sensitivity and reproducibility. The method was validated in different grain matrices. Limits of Quantitation (LOQ) of all mycotoxins were found between 0.1µg/kg and 5µg/kg. All LOQ meet the requirements of the GB methods. The accuracy of low, medium and high concentration spiked samples was between 80% and 120%. Matrix matched curves were used to quantify the unknown samples.

P-82 Chemical Fate Processes in Rice Field Ecosystems: Beyond the Regulatory Studies

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Rice culture offers unique environmental situations to investigate chemical processes not addressed in standard regulatory studies and not found in other cropping systems. The vast majority of rice fields are shallow water aquatic environments and sunlight plays a much greater role in fate processes then in deeper water bodies where the majority of sunlight is attenuated or scattered by suspended particulate matter. Secondary photodegradation processes including degradation by hydroxyl radicals can be highly significant as can degradation at sediment water interfaces. These processes have been shown to be highly significant to the degradation of the herbicide bensulfuron-methyl as well as the newer rice pesticides that have recently entered the market; they can also generate unique degradation products that may not be observed in other studies. In addition phototoxicity to aquatic organisms either residing in the rice field ecosystem or in aquatic environments receiving rice field tail waters is of potential concern and would not be addressed in the standard battery of tests conducted for pesticide registration. Recent work with the fungicide dicloran has shown that its presence at environmentally relevant concentrations can photochemically induce toxicity as well as sub-lethal impacts to crawfish, which is an economically important species in Louisiana and is harvested from rice fields. Measurements of a chemical's hydroxyl radical rate constant, fate in irradiated water-sediment systems and phototoxicity to ecologically relevant organisms can allow better assessments of chemicals used in rice.

P-83 Multiclass, multi-residue analysis of macrolides, polyether ionophores, tetracycline and sulfonamides by ultrahigh performance liquid chromatography tandem mass spectrometry

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An ultrahigh performance liquid chromatography tandem mass spectrometry (UHPLC-MS/MS) based multiclass and multi-residue method for the simultaneous analysis of six macrolides, five polyether ionophores, four tetracycline and seven sulfonamides in bovine and fish tissues was developed and validated. Sample extraction and clean-up were based on a modified QuEChERS (Quick, easy, cheap, effective, rugged, safe) extraction. Both matrix effect and uncertainties associated with sample preparation and instrumental analysis were minimised by the use of deuterated and carbon-13 labelled surrogate standards. Recoveries of analytes were generally satisfactory and typically fell between 65 – 120 %. Both repeatability and intermediate reproducibility measured as relative standard deviations were in most cases less than 20 % (n = 6). The method displayed it *fitness for purpose* through satisfactory results obtained in an international proficiency scheme. The method was successfully applied for the simultaneous analysis of the four classes of veterinary drugs and antibiotics in bovine and fish tissues.

P-84 Analysis of Specific Organophosphorous Pesticides, Synthetic Pyrethroids, and 2,4-Dichlorophenoxyacetic acid by LC-MS/MS

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It is estimated that more than a billion pounds of pesticides are used in the United States yearly. Organophosphorous pesticides and synthetic pyrethroids are commonly used as pest control substances in agriculture, homes, gardens and for veterinary practices, while herbicides are used in agricultural, and around commercial and residential buildings to control weeds. It is therefore important to develop methods that are suitable for the evaluation of biological markers of human exposure. We developed a method to measure urinary metabolite concentrations of four organophosphorous insecticides, five synthetic pyrethroids and one herbicide. The method is based on enzymatic deconjugation of the analytes in urine, extraction and concentration using automated solid phase extraction followed by analysis using liquid

chromatography mass spectrometry. Detection of the analytes is performed by a triple quadrupole mass spectrometer with a heated electrospray ionization source in either positive or negative ion mode. Analytes are identified by their retention time, the precursor ion, two fragment ions, one for quantification (Q) and one for confirmation (C), and by the Q/C ratio. Isotopically labeled internal standards are used for precise and accurate quantification. The limits of detection range from 0.01-0.4 ng/mL, with mean accuracy calculated as spike recoveries of 86-106%. The intraday percent coefficients of variation ranged from 5-14%. The effects of freeze thaw cycles, 24 hour benchtop storage, and processed sample stability were also evaluated. This method is suitable for assessing human exposure to select non-persistent pesticides in large population studies such as the National Health and Nutrition Examination Survey.

P-85 Multi-Residue Method for Analysis of 480+ Compounds in Hemp Products

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Hemp, a variety of *Cannabis sativa*, is used as a source for fiber and oil and is thought to have medicinal properties. As the legalization of *Cannabis sativa* spreads across the US, hemp has become a popular ingredient in the dietary supplement industry resulting to a wide variety of products available to consumers. Therefore, it is becoming ever more important to analyze these products for contaminants, including pesticides.
Hemp is a challenging matrix to analyze using a multi-residue screen due to the 500+ naturally occurring compounds present within the plant. Standard pesticide screening methods like AOAC 2007.1, which utilizes the QuEChERS extraction using acetonitrile as extracting solvent followed by LC/MS/MS and GC/MS/MS can be used as a starting point for the analysis. Using commercially available hemp oil, we explored alternative solvents like methanol, acetone, and hexane for extraction. The hemp oil extracts were cleaned using different amounts of $MgSO_4$ and PSA. Additionally, other commercially available products such as VERDE, LipidX, and diatomaceous earth were used to clean the extracts. After comparison of different combinations of extraction solvent and clean-up procedures, we came up with a method that gave consistent acceptable recoveries on a range of hemp products.
The final method is a combination of acetonitrile as extraction solvent and clean-up that uses various ratios of $MgSO_4$ and PSA followed by LC/MS/MS and GC/MS/MS and covers 480+ compounds.

P-86 Determination of Chlorophenoxy and Other Acidic Herbicides in Food Commodities using Liquid Chromatography with Tandem Mass Spectrometry

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Additional development and expertise have been assembled in the pursuit of a single method to quantify acidic herbicides in various food commodities. Working from the progress established in our 2017 NACRW poster, additional improvements were made to the sample preparation and chromatographic portions of this analysis. Several additional herbicides, herbicide metabolites, and degradants have been included in the method. The matrix categories covered food groupings outlined by the SANTE/11813/2017 document, focusing on groups 1, 2, 3, 4a, 4b, 5, 6, and 8. Improvements were made to the extraction process, including using an increased volume of alkaline and acidified solutions to perform the hydrolysis of bound analyte forms. We additionally evaluated an optional mixed-mode SPE cleanup and concentration step to address the suppression observed in a QuEChERS based sample preparation procedure. The chromatography was also improved to reduce the turnaround time between injections and increase the response of the herbicides which require electrospray negative detection mode. Validation data and statistics generated from various matrices at spiking levels of 3, 10, and 50 ng/g will be presented.

P-87 Analysis of Furfuryl Alcohol in a Variety of Foods and Beverages

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Furfuryl alcohol is a natural byproduct formed by the Maillard reaction during the thermal processing of foods. Its potential ubiquity in foods and beverages, along with being considered a carcinogen, warranted development and validation of a method for identification and quantification of furfuryl alcohol in a variety of foods and beverages. A

simple extraction coupled with incorporation of stable labeled furfuryl-d5 alcohol as an internal standard was developed. Analysis was performed using ultra-high performance liquid chromatography – tandem mass spectrometry (UHPLC-MS/MS) using positive mode atmospheric-pressure chemical ionization (APCI+) detection. The method was validated in various food and beverage matrices with typical limit of quantitation of 1 ppm and recoveries ranging between 85-115% with RSDs <10%. The method is amenable to multiple matrix classes and could also include related furanic compounds 5-hydroxymethyl-2-furaldehyde and furfural if desirable.

P-88 Assessment of a Novel High Resolution MS Acquisition Procedure with Unique Informatics for Improved Contaminant Monitoring Selectivity and Simplified Data Review

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One of the biggest challenges associated with adopting high resolution mass spectrometry (HRMS) for routine regulatory contaminant monitoring is data interpretation. It takes a very experienced analyst to efficiently and reliably review the enormous amount of data produced for every sample. To monitor for both known and unknown analytes on HRMS systems, a data independent acquisition (DIA) procedure is typically used to generate precursor and product ions. Unfortunately, this approach makes it difficult to associate product ions with a specific precursor ion and creates a much higher background response compared to MS/MS transitions from a triple quadrupole system, leading to more challenging data review. One approach to address this issue uses an alternative acquisition procedure with a fixed quadrupole scan window (20 – 50 amu) to scan over the mass range of interest, alternating between low energy (for precursor ions) and a higher energy ramp (for product ions) in the collision cell. Finally, the TOF measures the precursor and product ions with equivalent mass resolution and duty cycle. The same algorithm used to filter HRMS data with ion mobility drift times may be applied to the data to isolate product ions that are associated with precursor ions that are in the quadrupole at the same "drift" time during a scan. This filtering of the data provides a pronounced spectral cleanup, generating characteristic product ion spectra for coeluting compounds whose precursor masses differ by at least ~5 amu. In this study, the proposed technique is applied to regulatory samples and compared with results from traditional LC-MS/MS and HRMS acquisition procedures.

P-89 Comparison of various data-acquisition modes in high resolution mass spectrometry (HRMS) for contaminants screening in aquacultured products

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Chemical contaminants in animal products such as fish are often of significant concern in food safety. Our lab has previously developed a wide-scope screening method to effectively monitor veterinary drug residues in aquacultured samples using LC with a quadrupole-Orbitrap HRMS, acquiring data in a full-scan MS in combination with all-ion-fragmentation (AIF) MS/MS mode. Recently, data-independent-acquisition (DIA) has emerged for use in food contaminants analysis. In this acquisition mode, a group of precursor (MS1) ions are introduced into the collision cell to produce product (MS2) ions which should theoretically provide increased sensitivity and selectivity along with better MS2 spectra compared to AIF for initial screening. In addition, the DIA acquisition mode allows for sequential changes in the defined width of isolation window across a mass range, which helps to utilize the mass detector more efficiently for the mass range where the analytes of interest are located. In the current study, we explore the capabilities of different data acquisition methods including non-targeted data independent analysis as well as targeted data-dependent MS^2 (DDMS) and parallel reaction monitoring (PRM) acquisition. We optimized the isolation width variables and acquisition parameters. The methods are evaluated with fish fortified with 70 test compounds to determine how many analytes can be detected and identified reproducibly. The fit-of-purpose of each acquisition mode is also evaluated by the quality of the MS/MS spectra and points across the peaks for the targeted compounds.

P-90 USEPA Method 625.1, New Method and New Instrumentation for Semivolatiles with SPE

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On September 27th 2017 USEPA method 625.1 was approved to allow the use of Solid Phase Extraction (SPE) for the extraction of industrial discharge samples. With the approval to extract with SPE a laboratory must demonstrate a validation study that includes all quality control criteria a proficiency testing sample as well as a matrix spike and matrix spike duplicate prepared with nine different discharges that must meet all Quality Control (QC) acceptance criteria. We will be discussing method 3535 Solid-Phase Extraction (SPE) utilizing the one-pass method. This novel way of extracting samples for USEPA 625.1 will be demonstrated by passing an acidified sample through a SPE disk and carbon cartridge to achieve the QC requirements and a nationwide validation package covering a full range of compounds in Tables 1 and 2. Compounds in Tables 1-3 will be demonstrated utilizing the one pass method with new SPE automation. The one pass method along with automated instrumentation will demonstrate the ability that disk SPE will handle industrial discharge samples as well as other challenging matrices that are encountered throughout environmental laboratories nationwide. The one pass method does result in less extraction time, solvent and eliminates matrix interfering emulsions that are quite common with USEPA 625.1 samples when the samples are basified.

P-91 Determination of anionic polar pesticides in various food matrices using a novel HILIC column chemistry by liquid chromatography-tandem quadrupole mass spectrometry

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Various multi-residue LC-MS/MS methods are available to analyze food for pesticide residues. However some pesticides are not amenable to such generic methods because they are highly polar and/or ionic in nature. Hence the extraction and retention under the generic C18 conditions becomes difficult. Common alternatives include derivatization, use of porous graphitised carbon columns and ion chromatography. However, due to time-consuming sample preparation, MS incompatible solvents and the need for specialized equipment and/or reagents, a simple approach, without derivatization, is preferred. Due to the physicochemical characteristics of these compounds, obtaining repeatable peak shape and robust methodologies can be challenging.
A simple UPLC-MS/MS method will be presented for the determination of highly polar, anionic pesticides, which provides analyte retention and excellent sensitivity, robustly, to exceed enforced MRLs. A panel of representative anionic polar pesticides, including aminomethylphosphonic acid (AMPA), glufosinate and glyphosate have been targeted in a selection of relevant foodstuffs prepared using a modified version of the Quick Polar Pesticides (QuPPe) extraction method. Chromatographic separation was achieved on a novel hydrophilic interaction liquid chromatography (HILIC) column, applying gradient method.
The developed chromatographic method was applied to analyze glyphosate and other polar pesticides in spinach, lentils and wheat. Method performance was evaluated, in the absence of isotopically labelled internal standard, by assessing chromatographic performance, sensitivity, calibration characteristics and repeatability. Satisfactory results were obtained for all studied analytes in three matrices and will be reported.

P-92 Determination of fipronil and its metabolite fipronil sulfone in eggs by LC-MS/MS using a modified QuEChERS method

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In late July 2017 millions of eggs in Europe and Asia were withdrawn from sale due to an alert from Belgian officials via the EU RASFF portal. Fipronil had been found in eggs produced by some Dutch farms at concentrations above the EU maximum residue level. Fipronil is an insecticide intended for professional pest control to combat infestation of insects as well as in veterinary medicine to combat fleas, mites, and ticks on home animals. It is a highly toxic compound and it is not authorized for use around food producing animals. In this work we present a rapid and effective LC-MS/MS method for determination of fipronil and its metabolite fipronil sulfone in eggs after extraction using a modified version of QuEChERS. A pass-through SPE stage provided effective removal of co-extracted lipids.
Results from validation of the method demonstrated excellent performance for the detection, identification and

quantification of fipronil and fipronil sulfone in egg, meeting the criteria recommended in the guidance document SANTE/11813/2017. Excellent sensitivity, selectivity and quantitative performance using matrix-matched calibration were observed. Mean recovery and repeatability (RSD) for fipronil and fipronil sulfone from measurements of spikes at 0.002 mg/kg and 0.02 mg/kg was 95 % (1.2 % RSD) and 96 % (1.4 % RSD), respectively. The efficiency of the pass through SPE cleanup was confirmed by the absence of matrix effects and the removal of at least 95 % of phospholipids.

P-93 Improved SPE for LC-MS Determination of Ractopamine and Other Beta-Agonist Drugs in Tissue Samples

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Beta-agonist (beta-andronergic) drugs are used in animal husbandry as agents for growth enhancement and to promote leanness. Among these drugs, ractopamine and zilpaterol are approved for use in the USA and Canada for cattle, pigs, and turkeys. These and other beta-agonist drugs are not allowed for such use in the EU and in much of the rest of the world. Reliable analytical methods are necessary to determine residues of these compounds in samples obtained from animals raised for human consumption. These are challenging samples for residue analysis because they are high in fat and phospholipids, co-extracted substances that can lead to interference in the UPLC-MS analysis or contamination of sensitive instrumentation. The USA tolerance level for ractopamine is 30 µg/kg (ppb) in bovine muscle or 90 µg/kg in liver. Therefore, there is a need for reliable analytical methods for residue levels in the low µg/kg range. However, there is increasing interest in monitoring for illegal use of ractopamine and other beta-agonists in tissue samples with no measurable residue allowed. In this study, after a simple and fast solvent extraction, a novel and rapid SPE cleanup protocol was employed for effective removal of phospholipids and other co-extracted substances prior to LC-MS analysis. The goal was to obtain detection limits suitable for the monitoring of illegal use of these veterinary drugs.

P-94 Determination of acidic herbicides in water using liquid chromatography-tandem quadrupole mass spectrometry

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A range of different acidic herbicides are widely approved for use in agriculture, recreational areas and watercourses worldwide. Herbicides can enter water bodies either directly through the spray and spray drift or indirectly via surface water run-off or leaching and sub-surface draining. In the aqueous environment, residues of acidic herbicides are most commonly found in the form of the free acid.

The presence of pesticides in water is regulated in Europe through different directives. The Drinking Water Directive sets a maximum limit of 0.1 µg/L for individual pesticide residues present in a sample. The Water Framework Directive (WFD) deals with surface waters, coastal waters, and groundwater. Member States must identify River Basin Specific Pollutants and set their own national environmental quality standards (EQS) for these substances.

In order to meet the regulatory limits, these compounds are traditionally extracted from sample and concentrated by SPE followed by either LC-MS/MS or GC after derivatization of analytes. This work shows the performance of a method for the determination of 20 acidic herbicides by direct large volume injection of water samples by UPLC-MS/MS. The method is simple, fast, and reliable, which also avoids the time, costs and potential losses associated with SPE. Calibration characteristics, linearity and residuals were very good over the concentration range studied and accuracy of the method was shown to be excellent. The results of the validation indicate that the method is suitable for the determination of acidic herbicides in both drinking and surface waters for monitoring purposes.

P-95 Determination of chlorate and perchlorate using a novel HILIC column chemistry by liquid chromatography-tandem quadrupole mass spectrometry

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Residues of chlorate and perchlorate detected in plant-based foods can be traced to sources such as from irrigation water contaminated with rocket solid propellants and fertilizers and the use of chlorinated water during crop washing or

disinfection of surfaces during food production. Chlorate was used as a herbicide, which although no longer approved, was still covered by pesticide residues regulations so the default MRL of 0.01 mg/kg applied. However, concentrations from levels found in foods were often above this limit so proposals have been made to introduce MRLs for chlorate in foods at levels based on occurrence data (0.01 to 0.7 mg/kg). There are currently no regulatory maximum limits for perchlorate in food in Europe but the European Commission did introduce a range of reference levels for intra-Union trade (0.1 to 1.0 mg).

Chlorate and perchlorate have traditionally been analyzed by ion chromatography, requiring the use of specialized equipment and more recently by LC-MS/MS, using porous graphized carbon or "mixed mode" columns. Here we present an alternative LC-MS/MS method using a novel hydrophilic interaction liquid chromatography (HILIC) column and MS-friendly mobile phase.

Several commodities have been analysed using this method, after extraction using the QuPPe method. The chromatography (retention, separation and peak shape), sensitivity, calibration characteristics and repeatability of the method was shown to be excellent and to meet the requirements in the SANTE guidelines (Document 11813/2017). Incurred residues of both chlorate and perchlorate, detected in a number of samples, were quantified using the standard addition functionality of the software.

P-96 Rapid Evaporative Ionization Mass Spectrometry for high throughput screening in food analysis: the good and bad and the smelly

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Rapid Evaporative Ionisation Mass Spectrometry (REIMS) combined with multivariate statistical analysis (Principal Component Analysis and Linear Discriminate Analysis) is an emerging technique for near real time characterization of tissues with no requirement for sample preparation.
Due to their high market value, fish are often targets for species substitution, adulteration, or mislabeling. REIMS has been successfully used as a fast profiling technique capable of achieving accurate species identification without the need for any sample preparation. Significant time comparisons between REIMS and polymerase chain reaction (PCR) were observed when analyzing 6 mislabeled samples demonstrating how REIMS can be used as a complimentary technique to detect fish fraud.
The utility of REIMS has also been explored for the direct analysis of meat samples from livestock treated with illegal growth promoters. The REIMS analysis generated specific lipid profiles which enabled differentiation of meat samples collected from pigs treated with ractopamine via their feeding regime.
Boar taint is a contemporary off-odor present in meat of uncastrated male pigs. As surgical castration of pigs is to cease in Europe by 2018, there is a lot of interest in rapid tests that can screen out tainted carcasses without reliance on sensory analysis. Screening of samples of neck fat using REIMS enabled discrimination between sow, tainted and untainted boars.
These case studies demonstrate that REIMS implemented in an untargeted-metabolomics workflow can be considered as a high-throughput and accurate strategy for real-time classification of fish and meat samples in relation to issues relating to food authenticity, safety and quality.

P-97 Improved Analysis of Propylene Oxide, Propylene Chlorohydrin and Propylene Bromohydrin

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The importance of propylene oxide (PPO) treatments for stored product protection has only increased in recent years, especially as the implementation of FSMA in the US puts pressure on tree nut producers to pasteurize their product. Unfortunately, analysis is complicated by the ease with which PPO will undergo nucleophilic reaction with water to form propylene glycol, or with chloride and bromide to form propylene chloro and bromo- hydrin, which can artificially lower the detected PPO residue. Avoiding the formation of these halohadrins (PXH) is of particular importance as they

face regulatory scrutiny as carcinogens. The benefits and deficiencies of several methods of analysis for PPO and PXH, including the aqueous extraction used in ASTA method 23.1 and the MTBE extraction method previously reported by the authors, will be discussed. Novel methods utilizing dynamic headspace extraction and solid phase microextraction (SPME) will also be reported with particular emphasis on preventing artefactual effects. Preliminary experiments have demonstrated that while headspace sampling methods can significantly improve sensitivity for PPO, PCH and PBH (~3 orders of magnitude decrease in LOD for PBH), great care must be taken to avoid artefactually raising PCH and PBH levels. The use of autosamplers (either dynamic headspace or L-PAL3 with SPME attachment) can greatly reduce injection to injection variability and reduce the number of person-hours required for analysis, but to fit walnuts or almonds into headspace vials they must be chopped or ground exposing further chloride or bromide to react with PPO. Experiments using manual SPME sampling will allow the use of glassware that can accommodate whole nuts. The use of iodide, or other nucleophiles, to compete with chloride and bromide for the reaction with PPO will also be examined.

P-98 Simplified Sample Preparation: Residue Analysis of Wastewater and Ground Water by LC-MS/MS

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Residue analysis of wastewater is becoming more important and more varied. Regulatory requirements are in place for environmental, wastewater, raw & finished water quality for home, agriculture and industrial uses. Groundwater supplies drinking water for 51% of the total U.S. population and 99% of its rural population. Unfortunately, groundwater is susceptible to pollutants due to the widespread use of pesticides and fertilizers. With the current increase in drug abuse, there is an increase in drug metabolites and creatinine in wastewater. Traditionally, syringe filtration or centrifugation have been used to remove particulates and reduce possible matrix interference prior to LC/MS analysis. However, these techniques are time consuming, adversely impact reproducibility and quantification. We investigated the potential for streamlining sample preparation method for residue analysis in ground water and city wastewater using the Thomson Filter Vials.

P-99 Direct Immersion Solid-Phase Microextraction Analysis of Multi-class Contaminants in Edible Seaweeds by Gas Chromatography-Mass Spectrometry

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The dietary value of edible seaweeds has prompted their consumption as a healthy food worldwide and also led to the development of seaweed-based industries in recent years. However, the Maximum Residue Levels (MRLs) for hazardous residues in edible seaweeds are still not fully established and regulated. Solid-phase Microextraction (SPME) was employed in this work as an alternative to liquid-liquid extraction (LLE), for extraction of PAHs, PCB, and pesticides. A matrix compatible coating, namely PDMS/DVB/PDMS, was used for the direct immersion (DI)-SPME in dry seaweed samples. As the target contaminants possess a wide range of physical-chemical properties, multivariate experimental design was used for method optimization. Optimum conditions suitable for simultaneous quantitation of all targeted compounds, namely buffer at pH=7.0, 20% acetone (v/v), 10% NaCl (w/w), 0.02% NaN3, 60 min DI extraction at 55 °C, and 20 min desorption at 270 °C, afforded limits of quantitation (LOQs) in the range of 1-30 µg kg-1, a wide linear range of 5-2000 µg kg-1, the attainment of satisfactory determination coefficients (R2>0.99) with no significant lack of fit (p>0.05) at the 5% level, and satisfactory accuracy and precision values. The established method was then used for screening of commercial, edible dry seaweeds, with PCBs (≤16.0 ng g-1) and PAHs (≤15.5 ng g-1) detected in some samples. This method overcomes most challenges commonly encountered in dry sample analysis applications and represents the first report of a DI-SPME method employing the matrix-compatible fiber for simultaneous multiclass and multiresidue analysis of seaweeds.

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