54th Annual

NACRW

NORTH AMERICAN CHEMICAL RESIDUE WORKSHOP

Naples Grande Beach Resort
Naples, Florida
July 23-26, 2017

"Bringing Scientists Together to Develop and Validate Better Methodologies"

Cover designed by UCT, Inc.

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FUTURE MEETING DATES

2018   July 22-25
Naples Grande Resort

2019   July 21-24
Naples Grande Resort
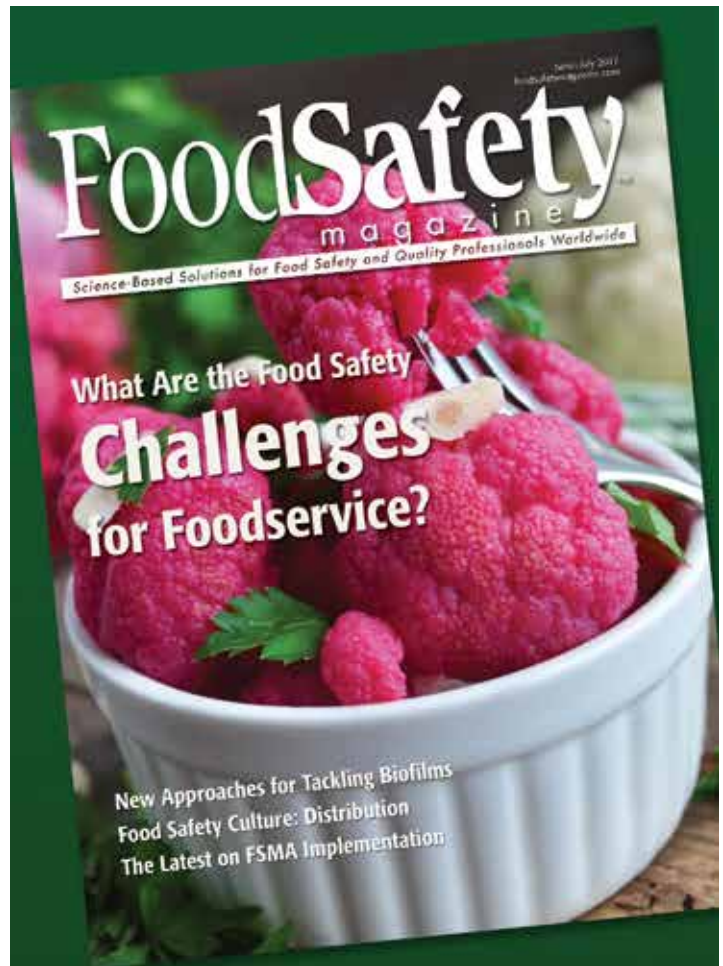
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Dear Attendees, Exhibitors, Sponsors and Guests;

Welcome everyone to the 54th, North American Chemical Residue Workshop! 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 year after year!

For those ready to dive into the science upon arrival, we are offering a one-day short course on Sunday. The course, *Efficient Start-to-Finish Analysis of Chemical Residues,* will provide students with a thorough understanding of chemical residue testing, starting from sample processing to data handling. The instructor, Dr. Steve Lehotay is a NACRW Excellence Award winner and world renown leader in the field of chemical residue analysis. We are grateful to have him teach these important concepts to class participants.

For those interested in some friendly competition, we've obtained a discounted rate at the Naples Grande Golf Club. Tee times can be scheduled prior to or following the workshop. We hope you will make plans to attend the welcome reception on Sunday evening, open to all our attendees. This is great opportunity to visit with NACRW friends and network with our exhibitors. Since this is the first year at the Naples Grande, we invite you to attend our Monday evening on-site social event! We've planned a Hawaiian-style luau, with great food and plenty of opportunities to meet new friends and re-connect with old acquaintances. We hope that you will take advantage of some or all the social opportunities available at the workshop!

For the third year in a row, FLAG Works, Inc. is sponsoring the NACRW Excellence Award. The award topic is Excellence in Separation Science Relevant to Chemical Residue Analysis. We are pleased to announce the recipient of the 2017 NACRW Excellence Award is Harold McNair, Professor Emeritus in Analytical Chemistry from Virginia Polytechnic Institute and State University. During his distinguished career, Dr. McNair has authored over 200 peer reviewed journals, monographs, articles, books, and audio video publications pertaining to chromatographic separation science. He has presented, lectured, and taught seminars around the world for decades. Residue scientists around the world are indebted to Dr. McNair for his lifelong contributions in this field. Dr. Kevin Schug will be accepting the award and presenting on Dr. McNair's behalf. We are happy to have him at the workshop to highlight Dr. McNair's many achievements with the NACRW community.

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 Federal and State Regulatory Laboratories and the Mass Spectrometry Forum.

In addition to our oral sessions please attend the poster session, exhibitors, and vendor seminars. The posters 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 workshops starting on Sunday evening and occur 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 the fantastic volunteers who are on-site working hard to keep the workshop running smoothly. To the 2017 NACRW Organizing Committee, Program Committee, especially Yelena Sapozhnikova, Mark Crosswhite, 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,

Kelly Dorweiler, 2017 Organizing Committee President
Yelena Sapozhnikova and Mark Crosswhite, 2017 Program Committee Co-Chairs
2017 Organizing Committee and Program Committee members

NACRW and FLAG Works, Inc. dedicate this program to the memory of Wilma Fong

Wilma was known for her infectious smile, enthusiasm and readines to help anyone in need. Many remember her as a team player in many activities and the coach of the Westminister Oaks Table Tennis Team, which became the best in Tallahassee. Memories of Wilma will last in our hearts forever.

her loving husband George Fong
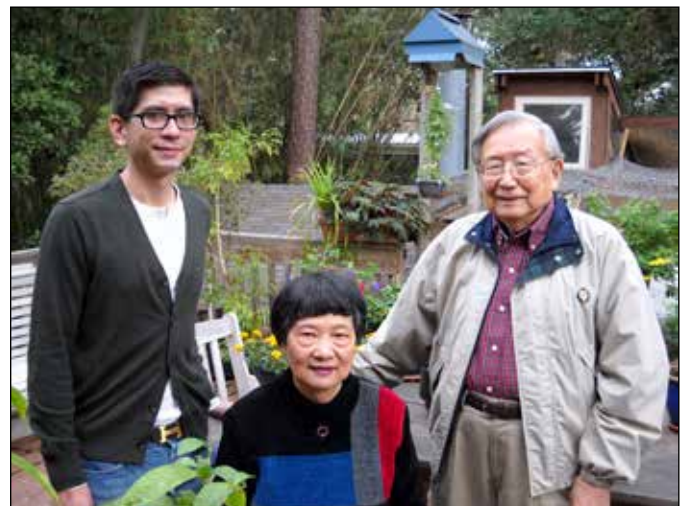
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The George and Wilma Fong Founders Award

In Appreciation for Years of Leadership and Dedication to the Florida Pesticide Residue Workshop and the North American Chemical Residue Workshop by Volunteering many hours over many years and who has worked to contribute to the Methodology and the Advancement of the workshop.

Past Recipients
2011 George and Wilma Fong-Founders
2012 Gail Parker
2013 Pat Beckett
2014 Sherry Garris
2015 Jack Cochran
2016 Amy Brown

FLAG Works, Inc. / 2017 North American Chemical Residue Workshop
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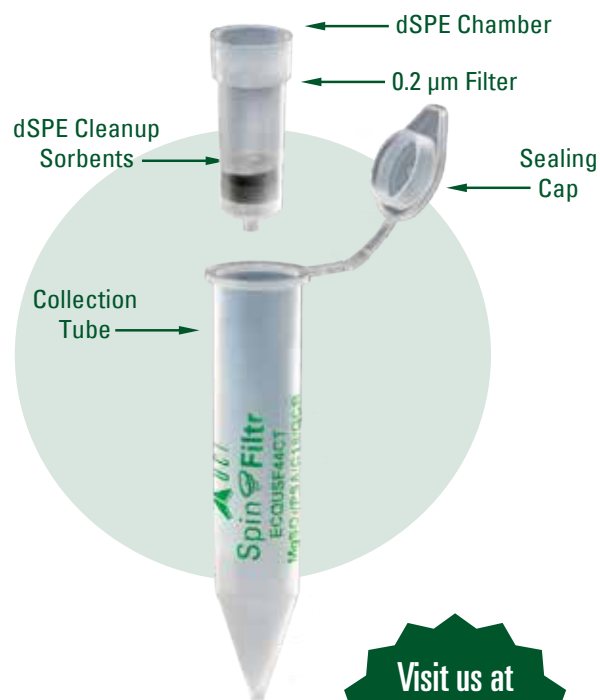
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Susan Genualdi, FDA

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Wendy Andersen, FDA
Katie Carlos, US FDA CFSAN
Jack Cochran, VUV Analytics
Jo Marie Cook, FDACS
Sara McGrath, FDA
Steven Moser, ODAFF
Paul Reibach, Smithers Viscient
Jian Wang, Canadian Food Inspection Agency
Paul Yang, Ontario Ministry of the Environment

Poster Committee

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

Poster Judges

Marc Engel, FDACS
Steven Lehotay, USDA ARS
Kate Mastovska, Covance Laboratories
Steven Moser, ODAFF
Yelena Sapozhnikova, USDA ARS
Robert Trengove, Murdoch University
Sheri Turnipsheed, US FDA/ORA/ADRC
Lawrence Zintek, US EPA

NAPLES GRANDE

BEACH RESORT

Hotel Overview

Meetings and Hotel

Vendor Seminars and Monday evening Social Event

Lobby Level

2nd Level

Exhibit Hall

General Session

Registration
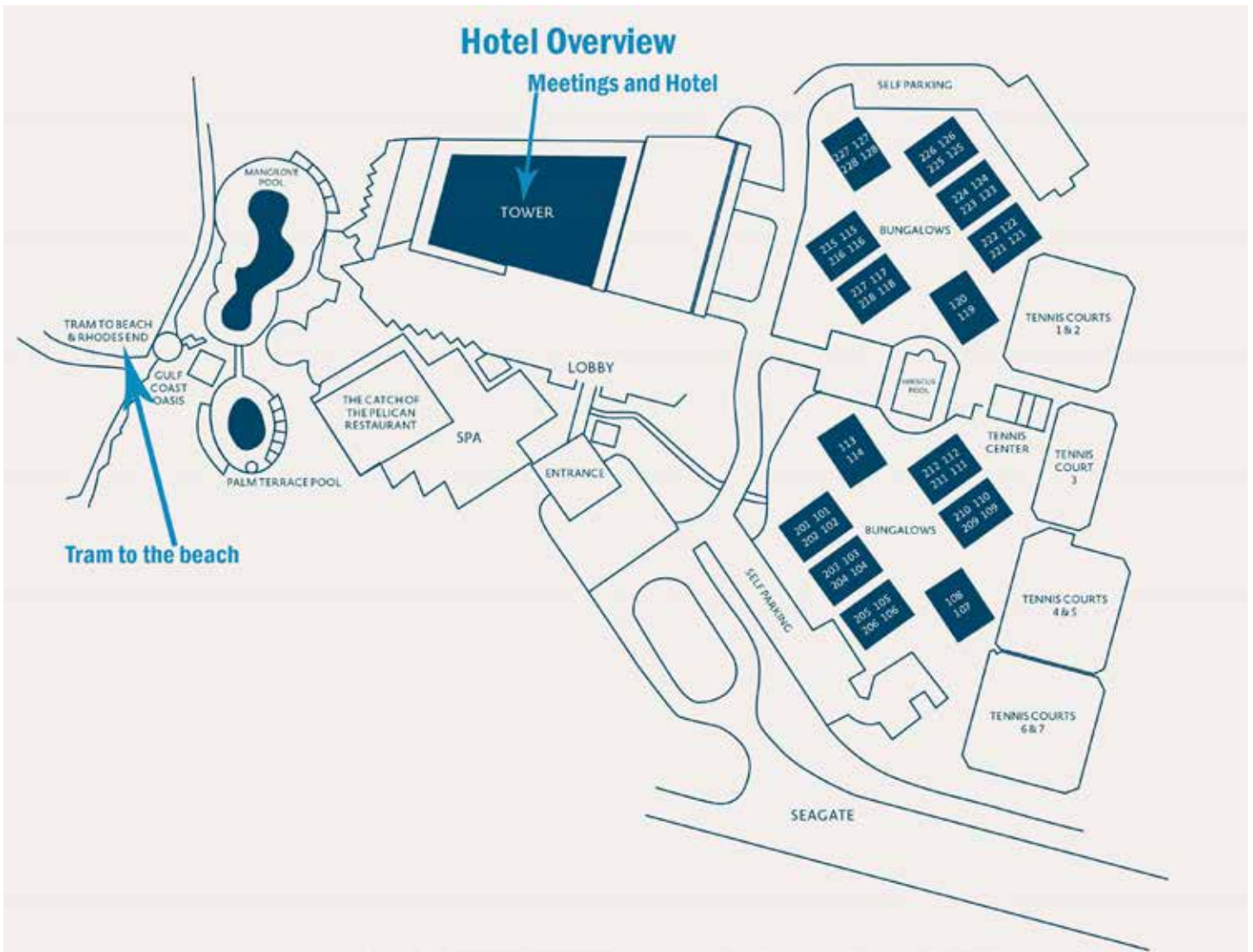
We would like to thank the following companies for their support of the 2017 NACRW

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NAPLES GRANDE
BEACH RESORT

MEETING AT A GLANCE

Sunday, July 23, 2017

8:00 am-4:00 pm	Short Course: Steven Lehotay	Acacia 4-6
	Efficient start-to-finish analysis of chemical residues	
1:00-5:00 pm	Exhibitor Setup	Royal Palm Ballroom
2:00-6:00 pm	Registration	Orchid Foyer
3:00-6:00 pm	Poster Board Set Up	Royal Palm Ballroom
4:00-5:00 pm	FDA/State Forum-government employees only	Banyan
5:15-5:45 pm	Moderator and Volunteer Training	Orchid Ballroom
6:15-7:15 pm	Restek Vendor Seminar	Vista Ballroom, lobby level
7:30-9:30 pm	Welcome Reception	Royal Palm Ballroom

Monday, July 24, 2017

All Day	Registration	Orchid Foyer
7:00-10:00 am	Poster Board Set Up	Royal Palm Ballroom
7:30-8:15 am	Early Morning Coffee	Orchid Foyer
7:15-8:15 am	Waters Corporation Vendor Seminar	

8:30-8:40 am	**Opening Remarks**	**Orchid Ballroom**
	Sherry Garris, Chair, FLAG Works, Inc.	
8:40-8:45 am	**Introduction and Presentation of NACRW Excellence Award**	
	Kelly Dorweiler, 2017 NACRW President	
8:45-9:30 am	**Presentation by Excellence Award Winner**	
9:30-10:45 am	**SESSION 1:**	**Orchid Ballroom**
	Advanced Detection Techniques	

10:45-noon	Exhibition and Poster Opening	Royal Palm Ballroom
11:00-noon	Poster Session A (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 Corporation Vendor Seminar	Vista Ballroom, lobby level

1:30-3:10 pm	**SESSION 2:**	**Orchid Ballroom**
	Veterinary Drugs and Anti-Microbial Resistance	
3:10-3:55 pm	**BREAK (Exhibition & Posters)**	**Royal Palm Ballroom**
3:55-5:35 pm	**SESSION 3:**	**Orchid Ballroom**
	Novel and Emerging Food Contaminants	
6:30-9:30 pm	**Luau Social Event - Naples Grande Beach Resort**	**Sunset Veranda/Vista Ballroom**

Tuesday July 25, 2017

All Day	Registration	Orchid Foyer
All Day	Exhibition & Posters	Royal Palm Ballroom
7:30-8:15 am	Early Morning Coffee	Royal Palm Ballroom
7:15-8:15 am	Phenomenex Vendor Seminar	Vista Ballroom, lobby level

8:30-10:45 am	**SESSION 4:**	**Orchid Ballroom**
	Advanced Sample Preparation	

MEETING AT A GLANCE

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	**Thermo Fisher Scientific Vendor Seminar**	**Vista Ballroom, lobby level**

1:30-3:10 pm	**SESSION 5:**	**Orchid Ballroom**
	State/Federal Laboratory Updates	

3:10-3:55 pm	**BREAK (Exhibition & Posters)**	**Royal Palm Ballroom**

3:55-5:00 pm	**SESSION 6:**	**Orchid Ballroom**
	Mass Spectrometry Forum	

5:05-6:00 pm	**Organization Committee Meeting**	**Orchid Ballroom**
	open to all attendees	

6:00-10:30 pm	**Shuttle service to/from 5th Avenue downtown Naples**	**Outside - Hotel Entrance**
	Two shuttles scheduled to run every 15 minutes	

Wednesday, July 26, 2017

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	**SCIEX Vendor Seminar**	**Vista Ballroom, lobby level**

8:30-10:45 am	**SESSION 7:**	**Orchid Ballroom**
	Natural Products, Supplements, and *Cannabis*	

10:45-noon	**BREAK (Exhibition & Posters)**	**Royal Palm Ballroom**
12:00-1:00 pm	**Agilent Technologies Vendor Seminar**	**Vista Ballroom, lobby level**

1:05-2:45 pm	**SESSION 8:**	**Orchid Ballroom**
	Residue Analysis in Aquaculture Products and Water Systems	

2:45-3:15 pm	**BREAK**	**Orchid Foyer**
2:45-3:15 pm	**AOAC Pesticide Contaminant Sub-Committee**	**Orchid Ballroom**
	Meeting *- open to all attendees*	

3:15-4:55 pm	**SESSION 9:**	**Orchid Ballroom**
	General Topics	

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

6:00-10:30 pm	**Shuttle service to/from Mercato (Blue Martini)**	**Outside - Hotel Entrance**
	Two shuttles scheduled to run every 15 minutes	

Thursday, July 27, 2017

User Meetings

7:30-9:30 am	**SCIEX User Meeting**	**Royal Palm 1**
10:30 am-12:30 pm	**Agilent Technologies User Meeting**	**Royal Palm 3**

GENERAL INFORMATION

Registration
Check in once at the registration desk at your earliest opportunity
Sunday - 2:00 – 6:00 pm
Monday - 7:00 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 $100 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 to 9:00 pm and cash bar 9:00 to 9:30 pm
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 2017 NACRW. Please inform Yelena Sapozhnikova, 2017 Program Chair (yelena.sapozhnikova@ars.usda.gov), by August 31, 2017 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, 2017.

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

EXHIBITION HALL
AND
POSTER SESSIONS
Location: Royal Palm Ballroom, 2nd level

Exhibitors

AccuStandard Inc
Booth #42
www.accustandard.com

Advion, Inc.
Booth #34
www.advion.com

AFFINISEP
Booth #17
www.affinisep.com

Agilent Technologies
Booth #43 and 44
www.agilent.com

Analytical Sales and Services, Inc.
Booth #51
www.analytical-sales.com

Biotage
Booth # 39
www.biotage.com

BIPEA
Booth #14
www.bipea.org

Bruker
Booth #24
www.bruker.com

BUCHI Corporation
Booth # 29
www.buchi.com

Cambridge Isotope Labs
Booth # 30
www.isotope.com

Chem Service, Inc.
Booth # 31
www.chemservice.com

Crescent Chemical Co., Inc.
Booth # 38
www.creschem.com

Ellutia Inc
Booth # 50
www.ellutia.com

Gilson, Inc.
Booth # 55
www.gilson.com

Glas-Col, LLC
Booth # 46
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Booth # 33
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Horizon Technology Inc.
Booth # 9
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Indigo bioAutomation
Booth #15
www.indigobio.com/

J2 Scientific
Booth #28
www.j2scientific.com

LECO Corporation
Booth #1
www.leco.com

LGC Standards
Booth #53
www.lgcstandards.com/

MAC-MOD Analytical
Booth #64
www.mac-mod.com

MilliporeSigma
Booth #10
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www.pickeringlabs.com

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www.promochrom.com

Restek Corporation
Booth # 40
www.restek.com

SCIEX
Booth # 25 and 26
www.sciex.com

Shimadzu Scientific Instruments, Inc.
Booth # 35 and 36
www.shimadzu.com

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www.spexsampleprep.com

Teledyne Tekmar
Booth # 12
www.teledynetekmar.com

Thermo Fisher Scientific
Booth # 21 and 22
www.thermofisher.com

UCT, Inc.
Booth # 32
www.unitedchem.com

ULTRA Scientific
Booth # 49
www.ultrasci.com

USDA, FSIS, OPHS, (ALP)
Booth # 16
www.fsis.usda.gov

VICI DBS
Booth # 11
www.dbsinstruments.com/en/

Waters Corporation
Booth # 41 and 52
www.waters.com

SHORT COURSE

Sunday, July 23, 2017   8:00 am to 4:00 pm
Location: Acacia 4-6

PRE-REGISTRATION IS REQUIRED

"Efficient Start-to-Finish Analysis of Chemical Residues"

Instructor:
Steven Lehotay, PhD, Lead Scientist, USDA, Wyndmoor, PA

This 1-day educational event offers the chance for participants to learn about efficient high-quality and high-throughput analysis of different types chemical contaminants in any type of food, which are also applicable to other sample types. Analytes include pesticides, veterinary drugs, persistent organic pollutants, emerging and other contaminants. Just as a chain is only as strong as its weakest link, all aspects to the analytical process must be optimized and streamlined. Thus, efficient and effective ways to conduct sample processing (comminution), sample preparation (extraction and cleanup), analysis (separation and detection), and data handling (peak integration, quantification, and identification) will be described and discussed. A validated, automated approach to monitor hundreds of pesticides and environmental contaminants in foods by rapid, robust, and reliable GC- and LC- MS/MS techniques while avoiding human data review will be presented.

Agenda – a chronological list of the key sections of the course
1)   Introduction and Background: purposes for analysis, data quality objectives, and validation
2)   Sample Processing: theory and practice to quickly obtain minimal but representative sample test portions for analysis
3)   Sample preparation: theory and practice of different extraction and cleanup methods, including automation, to achieve high and consistent recoveries of a wide scope of analytes (or narrow scope depending on the application) while minimizing direct and indirect interferences and matrix effects from sample components
4)   Analysis: high-throughput methods less than 10 min each in gas and liquid chromatography coupled to modern mass spectrometric techniques for the detection of hundreds of analytes in complex matrices, or specialized contaminant applications such as nitrosamines or acrylamide
5)   Data handling: how to automatically integrate chromatographic peaks with reliable quantification and identification of analytes at trace levels in complex matrices without human review or manual re-integrations

Learning Outcomes – what are the key educational benefits of the course
With the appropriate instruments, the participant should be able to implement the approaches in their labs and applications described after taking this course. They will learn how to conduct high-throughput lab operations to obtain high-quality results in food monitoring applications.

You are warmly invited to 12th European Pesticide Residue Workshop.

Join Europe's leading meeting for the latest concepts and developments in the field of pesticides in food and drink. Exchange information and experience and connect to experts from governmental and commercial laboratories, public authorities, regulatory bodies, food producers, processors, retailers, agrochemical manufacturers and other interested parties from all parts of the world. All important vendors of analytical equipment and consumables will present their latest equipment for residue analysis in a large exhibition area.

In the city of 'Oktoberfest' you will enjoy the Bavarian hospitality.

We are looking forward to welcome all of you in Munich for EPRW 2018!

Contact:
EPRW 2018
c/o Interplan AG
E-Mail: eprw2018@interplan.com

For more information, please visit:
www.eprw2018.com

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 23, 2017, 6:15 p.m. to 7:15 p.m.   **RESTEK**
Location: Vista Ballroom, Lobby Level

Soft Solutions to Hard Problems: Saving Time and Money in the Laboratory with the *EZ*GC Web Tools
Jonathan Keim, Chris Rattray, Chris English
Restek Corporation, 110 Benner Circle, Bellefonte, PA 16823, USA

Analytical laboratories face continual pressure to increase productivity while decreasing costs. With recent advances in available technology, focus on optimizing analytical methods, and optimization of key instrument parameters, we can find valuable opportunities to save time and money. In this talk, we will share work showing how the *EZ*GC suite of online tools can be used to improve gas chromatographic analyses.

We will show several examples of how the free software can be used for method development. Additionally, these tools can be used to improve your analysis in various ways such as maintaining high quality separations with much shorter columns, the importance of optimizing your splitless valve time, and switching carrier gasses.

V-2 Monday, July 24, 2017, 7:15 to 8:15 am   **WATERS CORPORATION**
Location: Vista Ballroom, Lobby Level

Part 1
Overcoming the Challenges of Analysing Polar Pesticides in Food
Dimple Shah, Senior Scientist, Waters Corporation

A panel of representative polar pesticides has been targeted in a single LC-MS/MS method. Extracts of various foodstuffs were prepared in accordance with the Quick Polar Pesticides (QuPPe) extraction method. Chromatographic separation was achieved on mixed mode / hydrophilic interaction liquid chromatography (HILIC), applying a mobile phase gradient. Chromatographic performance was evaluated in accordance with SANTE guideline document 11954/2015, certain limitations were determined for the challenging analysis of these highly polar pesticides. Overall method performance was evaluated by assessing linearity, accuracy and sensitivity.

Part 2
Fast CAN be clean! Food Sample Cleanup Using Oasis PRiME HLB

Jeremy Shia, Product Manager, Waters Corporation

It is very challenging to develop specific sample preparation protocols for multiresidue contaminant analysis due to the complexity of food matrices. Better but potentially time-consuming cleanup procedures using traditional solid phase extraction were substituted by simple techniques with generic cleanup such as liquid-liquid extraction or protein precipitation. Consequently, insufficient generic cleanup contributes to the increased instrument maintenance and system downtime. Oasis PRiME HLB can efficiently remove common interferences

such as fats, phospholipids and pigments from food matrices. No method development is required by following the recommended pass-through protocol.   The cleanup step is fast and easily integrated following the initial extraction step but gives cleaner extracts compared with generic cleanup protocols. In this seminar, we will demonstrate the use of this pass-through protocol for the challenging food samples. Better cleanup will result in better accuracy and robustness of the analytical method, and increased system uptime.

V-3 **Monday, July 24, 2017, 12:15 to 1:15 pm**   **LECO Corporation**
Location: Vista Ballroom, Lobby Level

What's Hiding in Your Hops: Identification and Quantitation of Pesticides in Hops Extracts with a Benchtop GC-TOFMS

Todd Richards, Joseph E. Binkley
LECO Corporation, 3000 Lakeview Avenue; Saint Joseph, MI 49085, USA; Todd_Richards@leco.com; Joseph_Binkley@leco.com

Due to their matrix complexity hops have traditionally been a difficult commodity to monitor for pesticide use. The enormous growth in the craft brewing sector along with the popularity of IPA style beers has led to an increased demand for hops. It is anticipated that the demand for routine pesticide monitoring will likewise continue to rise. In this study gas chromatography was combined with time-of-flight mass spectrometry (TOFMS) to detect commonly monitored pesticides as well as screen for new and emerging pesticides. Whole leaf hops and extracted hop resin were extracted using a combination of QuECHERs and solid phase extraction (SPE) cartridges.

The extracts were injected onto an Agilent 7890B gas chromatograph (GC) fitted with an Agilent Multimode Inlet (MMI) and a standard 30m x 0.25mm ID x 0.25μm Rxi-5SilMS column coupled to a LECO Pegasus BT-TOFMS. The data was collected at 20 spectra per second with a mass range of 35-520 m/z. Identification of incurred pesticides was established by matching deconvoluted spectra to standard MS libraries coupled with software integrated retention index (RI) screening.

Calibration curves were established for incurred pesticides and matrix matched standards were prepared for other commonly used hops pesticides. Statistically derived limits of detection (LOD) were calculated for pesticides in matrix and found to be below required reporting limits demonstrating the Pegasus BT-TOFMS to be an effective platform for screening and quantitation of pesticide residues in hops.

V-4 **Tuesday, July 25, 2017   7:15 to 8:15 am**   **PHENOMENEX**
Location: Vista Ballroom, Lobby Level

Chromatography Tools and Options for Improved Residues and Contaminants Testing

Scott Krepich, Phenomenex, Inc., 411 Madrid Avenue, Torrance, CA 90501, USA

The use of LC-MS/MS and Sample prep options such as QuEChERS and Solid Phase Extraction are commonly used in testing for residues and contaminants. Technological and intellectual advancements have helped us improve the use of current instrumentations and techniques to address the various challenges in food testing. Presented are techniques and tools to assist in improving chromatographic results as well as sample prep options for improved clean up and extraction of targeted analytes from various food matrices. Knowing what sample preparation and chromatographic options are available can help in determining the appropriate method for optimized results. Applications reviewed include: Polar and non-polar pesticides from produce and plant samples including cannabis, veterinary drugs, mycotoxins , and PFAS from drinking water and foods.

V-5 **Tuesday, July 25, 2017, 12:15 to 1:15 pm**   **THERMO FISHER SCIENTIFIC**
Location: Vista Ballroom, Lobby Level

New Products and Applications to Enhance Workflows for the Analysis of Residues and Contaminants

Richard Fussell[1], Ed George[2] and Paul Silcock[3]
[1] Thermo Fisher Scientific, Hemel Hempstead, UK; [2]Thermo Fisher Scientific, San Jose, CA, USA; [3]Thermo Fisher Scientific, Runcorn, UK

Confident Quantitation Workflows for Today and Tomorrow

Technical highlights of a newly introduced triple quadrupole MS platform will be discussed along with application data demonstrating sensitive, robust, reliable and reproducible detection and quantification of pesticides in leek. In addition, quantitation workflows for polar pesticides, and those of algal toxins in environmental water samples, will be presented. Also, workflow solutions such as the Thermo Scientific™ Pesticide Explorer Collection will be discussed along with software solutions that provide easy data review and reporting. For each application, excellent sensitivity, robustness, reliability and ease-of-use result in high quality data that enable confidence for every routine analytical testing laboratory.

Two Years On: The Impact of Orbitrap GC-MS in Food Analysis

In 2015, the first fully integrated Thermo Scientific™ Orbitrap™ GC-MS system became available. This technology has allowed new capabilities in both targeted and non-targeted GC-MS analysis by combining high resolving power, high mass accuracy, excellent sensitivity and linearity. Two years on, various scientists have explored the unique capability of this instrument in a diverse range of food-related applications. Highlighted examples will include pesticide residue analysis; persistent organic pollutants; food contact materials and authenticity analysis.

V-6 **Wednesday, July 26, 2017, 7:15 to 8:15 am**   **SCIEX**
Location: Vista Ballroom, Lobby Level

Potency, Terpenes and Pesticides Analysis Using High Resolution LC-MS/MS: Results and Comparison to Alternative Analytical Methodologies

Kris Chupka, Next Frontier Biosciences

Currently 26 states have some form of legalized cannabis. This emerging industry has substantial needs for accurate and reliable analytical testing. Next Frontier Biosciences uses high resolution LC/MS in a broad range of applications applied to raw materials and products. In this presentation, I will be discussing the development of applications and challenges along the way; for cannabinoid potency, residual pesticides and terpenes. I will also review comparable technologies and how these methodologies match up.

V-7 **Wednesday, July 20, 2016, 12:00 to 1:00 pm**   **AGILENT TECHNOLOGIES**
Location: Vista Ballroom, Lobby Level

Safeguard Our Foods with Technological Innovations

Dan-Hui Dorothy Yang [1], Theresa Sosienski [1], Phil Wylie [2] and Kai Chen [1]
[1]Agilent Technologies, Inc., Santa Clara, CA; [2] Agilent Technologies, Inc., Wilmington, DE

Chemical residues, either sprayed for crop production or generated by microorganisms, can be toxic if consumed in harmful doses. Regulatory agencies have set maximum residue levels (MRLs) for thousands of chemical-matrix combinations in food. The low MRL requirement in diversified matrices pose significant challenges to screen and quantify hundreds of analytes simultaneously. This work presents Agilent's key innovations on the Ultivo Triple Quad LC/MS and the 7250 GC/Q-TOF for targeted and untargeted analyses. For Ultivo, innovations such as the Cyclone Ion Guide and Vortex Collision Cell maximize quantitative performance in a much smaller analytical package. Instrument reliability are greatly enhanced with reduced interventions for maintenance. For the 7250 GC/Q-TOF, the improved ion path and detector electronics design provide better resolution and wider dynamic range. Available low energy EI workflows facilitate the untargeted study of contaminants. MassHunter Software provides a comprehensive package from data acquisition, method development and routine implementation.

Fruits and vegetables were evaluated for pesticides analysis, whereas grains, nuts and spices for mycotoxins. Matrices were prepared using Agilent QuEChERS kits and/or a modified EMR-Lipid kit. Most analytes could be detected below MRLs, highlighting the excellent sensitivity of the new instruments with good accuracy (80-120%) and precision (%RSD < 15%).

2017 NACRW EXCELLENCE AWARD
PRESENTED TO

Dr. Harold McNair (left) and former student,
Dr. Kevin Shug (right)

Dr. Harold McNair

A Teacher and Chromatographer Extraordinaire

Harold M McNair was born and raised in Miami, AZ. He was fortunate to receive a full scholarship (all expenses paid) to the University of Arizona, Tucson where he graduated in Chemistry, High Honors in 1955. He graduated in 1957 and 1959 with an MS in Electrochemistry and a Phd in Gas Chromatography from Purdue University. He built his own GC Instrument(1957); there were no commercial systems. He spent 1959 and 1960 on a Fullbright Fellowship in Holland when he worked with all the pioneers in GC, English and Dutch scientists - A.J.P. Martin(Nobel Prize), Marcel Golay (capillary columns) Professor A.I.M.Keulemans, built the second GC at Royal Dutch Shell Labs, Amsterdam. He worked on GC in industry for 8 years, Esso R&D, Linden, N.J.; H.P. Amsterdam and Varian Associates, Palo Alto, CA.

He came to Virginia Tech in 1968 as an Associate Analytical Professor where he taught and did research in Separation Science for 48 years. He served as Department Head for 2 years, 8 months in 1990. He supervised 61 graduate student theses, the research of 30 Post-doctoral Fellows and actively taught Separation Science and General Chemistry. He published over 8 books, many audio visual aids and over 150 research publications. He has received many research and teaching awards, including the Tswett Medal from the Russian Academy of Sciences, LCGC Life time Achievement Award and in 1993, he was one of the first 2 recipients of the Virginia Tech Alumni Teaching Award (out of a faculty of 1,200).

Dr. McNair is unable to attend, in his place former student, Dr. Kevin Shug will make the presentation on his behalf.

Dr. Kevin Shug

Kevin A. Schug is Professor and the Shimadzu Distinguished Professor of Analytical Chemistry in the Department of Chemistry and Biochemistry at The University of Texas at Arlington (UTA). He received his B.S. degree in Chemistry in 1998 from the College of William and Mary, and his Ph.D. degree in Chemistry from Virginia Tech in 2002 under the direction of Prof. Harold M. McNair. From 2003-2005, he performed post-doctoral research at the University of Vienna in Austria under the direction of Wolfgang Lindner. Since joining UTA in 2005, his research has been focused on the theory and application of separation science and mass spectrometry for solving a variety of analytical and physical chemistry problems, in the fields of environmental, pharmaceutical, biological, and energy research. He has over 125 peer-reviewed publications and 400 presentations, posters, and invited talks to his group's credit. He has been the primary mentor and research advisor to more than 20 graduate and 50 undergraduate students. Dr. Schug has received several research awards, including the 2009 Emerging Leader Award in Chromatography by LCGC Magazine and the 2013 American Chemical Society Division of Analytical Chemistry Young Investigator in Separation Science Award. For his teaching, he received the 2014 University of Texas System Regents' Outstanding Teaching Award and was named in 2016, as a Fellow of the University of Texas System Academy of Distinguished Teachers.

The title of Dr. Shug's presentation is:
"New Tools for the Determination of Chemical Constituents in Complex Matrices"

2017 - 54th ANNUAL NORTH AMERICAN CHEMICAL RESIDUE WORKSHOP

MEETING PROGRAM

Sunday, July 23, 2017

8:00 am-4:00 pm	**Short Course: Steven Lehotay**	**Acacia 4-6**
	Efficient start-to-finish analysis of chemical residues	

1:00-5:00 pm	Exhibitor Setup	Royal Palm Ballroom
3:00-6:00 pm	Poster Board Set Up	Royal Palm Ballroom
2:00-6:00 pm	Registration	
4:00-5:00 pm	FDA/State Forum-government employees only	Banyan
5:15-5:45 pm	Moderator and Volunteer Training	Orchid Ballroom
6:15-7:15 pm	**Restek Vendor Seminar**	**Vista Ballroom, lobby level**

7:30-9:30 pm	**Welcome Reception**	**Royal Palm Ballroom**

Monday, July 24, 2017

All Day	Registration	
7:00-10:00 am	Poster Board Set Up	Royal Palm Ballroom
7:30-8:15 am	Early Morning Coffee	Orchid Foyer
7:15-8:15 am	**Waters Corporation Vendor Seminar**	**Vista Ballroom, lobby level**

8:30-8:40 am	**Opening Remarks**	**Orchid Ballroom**
	Sherry Garris, Chair, FLAG Works, Inc.	

	NACRW Excellence Award Presentation and Keynote Address
8:40-8:45 am	**Introduction and Presentation of NACRW Excellence Award**
	Kelly Dorweiler, 2017 NACRW President

8:45-9:30 am	**Presentation by Excellence Award Winner**
	Kevin Schug, Department of Chemistry & Biochemistry, The University of Texas at Arlington, Arlington TX and Harold McNair, Department of Chemistry, Virginia Tech, Blacksburg VA
A-1	**New tools for the determination of chemical constituents in complex matrices**

9:30-10:45 am	**SESSION 1:**	**Orchid Ballroom**
	Advanced Detection Techniques	
	Chair: Paul Yang	

9:30-9:55 am	**Paul Zomer**, RIKILT Wageningen University & Research, Wageningen, The Netherlands
O-1	**The use of GC-Orbitrap-MS in multi-residue analysis of pesticides**

9:55-10:20 am	**Jack Cochran**, VUV Analytics, Cedar Park, TX, USA
O-2	**Gas chromatography - vacuum ultraviolet spectroscopy: a new tool for food analysis**

10:20-10:45 am	**Jon W. Wong**, U.S. Food and Drug Administration, College Park, MD, USA
O-3	**LC-HRMS, mass spectral libraries, and compound databases for screening chemical residues and contaminants**

10:45-noon	**Exhibition and Poster Opening**	**Royal Palm Ballroom**
11:00-noon	**Poster Session A (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 Corporation Vendor Seminar**	**Vista Ballroom, lobby level**

1:30-3:10 pm **SESSION 2:** **Orchid Ballroom**
Veterinary Drugs and Anti-Microbial Resistance
Chair: Fadi Aldeek

1:30-1:55 pm **Eric Verdon**, ANSES-Laboratory of Fougeres, Javené, France
O-4 **Approaches for the (broad) screening of veterinary drugs by full scan accurate mass determination**

1:55-2:20 pm **Thomas Bessaire**, Nestlé Research Center, Lausanne, Switzerland
O-5 **LC-MS/MS methods for an effective control of veterinary drugs in raw materials and manufactured products – a food industry perspective**

2:20-2:45 pm **Brian Veach**, U.S. Food and Drug Administration, Jefferson, AR, USA
O-6 **Rapid Fire mass spectrometry with enhanced throughput as an alternative to liquid-liquid salt-assisted extraction and LC-MS analysis for sulfonamides in honey**

2:45-3:10 pm **Charles Yang**, Thermo Fisher Scientific, San Jose, CA, USA
O-7 **Quantitative analysis with Orbitrap™ high-resolution LC-MS/MS and a new triple quadrupole LC-MS/MS for validation of a multi-residue veterinary medicine suite in cattle muscle**

3:10-3:55 pm **BREAK (Exhibition & Posters)** **Royal Palm Ballroom**

3:55-5:35 pm **SESSION 3:** **Orchid Ballroom**
Novel and Emerging Food Contaminants
Co-Chairs: Susie Genualdi and Sara McGrath

3:55-4:20 pm **Katherine Carlos**, U.S. Food and Drug Administration, College Park, MD, USA
O-8 **Determination of the bis(2-ethylhexyl) phthalate (DEHP) concentration of beer stored in bottles with PVC gaskets**

4:20-4:45 pm **Agustin Pierri**, Weck Laboratories, Inc., Industry, CA, USA
O-9 **LC-MS/MS determination of per- and poly-fluoroalkyl substances (PFAS) in eggs and dairy products**

4:45-5:10 pm **Anindya Pradhan**, Eurofins Central Analytical Laboratories, New Orleans, LA, USA
O-10 **A novel method for analysis of 3-monochloropropane-1,2-diol (3-MCPD), 2-monochloropropane-1,3-diol (2-MCPD) and glycidyl esters in oils and fatty foods using GC-MS/MS**

5:10-5:35 pm **Martin Dusek,** Research Institute of Brewing and Malting, Prague, Czech Republic
O-11 **A one-year experience with pesticide residue analysis in hops using QuEChERS based method: living up to expectations or blind alley?**

6:30-9:30 pm **Luau Social Event - Naples Grande Beach Resort** **Sunset Veranda/Vista Ballroom**

2017 - 54th Annual North American Chemical Residue Workshop

Tuesday July 25, 2017

All Day	Registration	
All Day	**Exhibition & Posters**	**Royal Palm Ballroom**
7:30-8:15 am	**Early Morning Coffee**	**Royal Palm Ballroom**
7:15-8:15 am	**Phenomenex Vendor Seminar**	**Vista Ballroom, lobby level**

8:30-10:45 am   **SESSION 4:**   **Orchid Ballroom**
Advanced Sample Preparation
Chair: Jo Marie Cook

8:30-8:50 am   **Paul Reibach**, Smithers Viscient, Wareham, MA, USA
O-12   **Incurred residues - what are they and how much is present in our food supply?**

8:50-9:10 am   **Steven Lehotay**, USDA Agricultural Research Service, Wyndmoor, PA, USA
O-13   **How low can we go? Study to assess the accuracy of different test sample weights in pesticide residue analysis of fruits and vegetables using different chopping methods**

9:10-9:30 am   **Manol Roussev**, Wessling Group, Berlin, Germany
O-14   **Advantages of cryogenic milling using liquid nitrogen for the routine analysis of pesticide residues in food and feed**

9:30-9:50 am   **Rob Gooding**, BASF, Research Triangle Park, NC, USA
O-15   **Utilization of 96-well plates in residue analysis. Micro-extraction is a fast, cost effective, and green technology to determine residue in various matrices**

9:50-10:10 am   **Johannes Corley**, Self-employed, East Brunswick, NJ, USA
O-16   **Isotopically labelled compounds, nuclear and isotopic techniques in food safety monitoring**

10:10-10:45 am   **Question and Answer Forum. Expert panel:** JoMarie Cook, Steven Lehotay, Paul Reibach, Manol Roussev, Rob Gooding, Johannes Corley

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	**Thermo Fisher Scientific Vendor Seminar**	**Vista Ballroom, lobby level**

1:30-3:10 pm   **SESSION 5:**   **Orchid Ballroom**
State/Federal Laboratory Updates
Co-Chair: Katherine Carlos and Steven Moser

1:30-1:55 pm   **Brittany Holmes**, WSDA, Chemical & Hop Lab, Yakima, WA, USA
O-17   **Chemical & Hop Laboratory - 65 years of accomplishments and continuing challenges**

1:55-2:20 pm   **Marc Engel**, Florida Department of Agriculture and Consumer Services, Tallahassee, FL, USA
O-18   **Predicting mercury exposure to women of child-bearing age from various Grouper consumption scenarios**

2:20-2:45 pm   **Narong Chamkasem**, U.S. Food and Drug Administration, Atlanta, GA, USA
O-19   **Analysis of polar pesticides using a mixed-phase mode HPLC column**

2:45-3:10 pm   **Eugene Chang**, US Food and Drug Administration, Irvine, CA, USA
O-20   **An ion pair reverse-phase liquid chromatography method coupled to tandem mass spectrometry for analysis of glyphosate and its metabolites**

| 3:10-3:55 pm | BREAK (Exhibition & Posters) | Royal Palm Ballroom |

| 3:55-5:00 pm | SESSION 6:
Mass Spectrometry Forum
Chair: Brad Barrett and Brian Eitzer | 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 5th Avenue downtown Naples
Two shuttles scheduled to run every 15 minutes | Outside - Hotel Entrance |

Wednesday, July 26, 2017

Until noon	Registration	
Until noon	Exhibition & Posters	Royal Palm Ballroom
7:30-8:15 am	Early Morning Coffee	Royal Palm Ballroom
7:15-8:15 am	SCIEX Vendor Seminar	Vista Ballroom, lobby level

| 8:30-10:45 am | SESSION 7:
Natural Products, Supplements, and *Cannabis*
Co-Chairs: Paul Reibach and Jack Cochran | Orchid Ballroom |

| 8:30-8:55 am
O-21 | **Rick Jordan**, Pacific Agricultural Laboratory, Sherwood, OR, USA
Seeing the trees through the forest – analytical challenges and strategies for residue analysis in *Cannabis* |

| 8:55-9:20 am
O-22 | **Heather Krug**, Colorado Department of Public Health and Environment, Denver, CO, USA
Cannabis pesticide residue testing implementation: The Colorado experience |

| 9:20-9:45 am
O-23 | **Paul Reibach**, Smithers Viscient, Wareham, MA, USA
Pesticide residues in *Cannabis*: pesticide exposure risk assessment |

| 9:45-10:10 am
O-24 | **Kevin Armbrust**, Louisiana State University, Baton Rouge, LA, USA
The roles of national associations in state and federal regulatory cooperation: implications for future *Cannabis* policy |

| 10:10-10:45
O-25 | **Jana Hajslova,** University of Chemistry and Technology, Prague, Czech Republic
Challenging analytical strategies to characterize the cocktail of chemicals occurring in *Cannabis* plants and products thereof |

| 10:45-noon | BREAK (Exhibition & Posters) | Royal Palm Ballroom |
| 12:00-1:00 pm | Agilent Technologies Vendor Seminar | Vista Ballroom, lobby level |

| 1:05-2:45 pm | SESSION 8:
Residue Analysis in Aquaculture Products and Water Systems
Chair: Wendy Andersen | Orchid Ballroom |

| 1:05-1:30 pm
O-26 | **Estelle Dubreil**, ANSES-Laboratory of Fougeres, Javené, France
Dye residues in aquaculture products: targeted and metabolomics mass spectrometric approaches to track their abuse |

1:30-1:55 pm O-27	**Ryan Gibbs**, Canadian Food Inspection Agency, Dartmouth, NS, Canada **A multi-class method for the determination and confirmation of veterinary antibiotics and growth promoters in aquaculture products by LC-MS/MS and LC-HRAM-MS**
1:55-2:20 pm O-28	**Yelena Sapozhnikova,** USDA Agricultural Research Service, Wyndmoor, PA, USA **Analysis of pesticides, POPs and emerging contaminants in catfish**
2:20-2:45 pm O-29	**Simon Hird,** Waters Corporation, Wilmslow, Cheshire, UK **Targeted and non-targeted approaches to the determination of microcystins and other cyanotoxins in water using combinations of one and two-dimensional liquid chromatography (1D & 2D LC) and tandem quadrupole (TQ), quadrupole time of flight (QTof) and ion mobility quadrupole time of flight (IMS QTof) mass spectrometry**

2:45-3:15 pm	**BREAK** **Orchid Foyer**
2:45-3:15 pm	**AOAC Pesticide Contaminant Sub-Committee Meeting** *- open to all attendees* **Orchid Ballroom** **Co-Chairs: Steven Moser and Ping Wan**
3:15-4:55 pm	**SESSION 9:** **Orchid Ballroom** **General Topics** **Chair: Brian Eitzer**

3:15-3:40 pm O-30	**James Edwards**, Indigo BioAutomation, Inc., Indianapolis, IN, USA **Software Automation Strategies for Improving Quality and Throughput of Highly Complex LC-MS/MS Analysis**
3:40-4:05 pm O-31	**Paul Yang**, Ontario Ministry of the Environment, Toronto, Canada **Effect-directed analyses of endocrine disrupting compounds using liquid chromatography-mass spectrometry and ER-CALUX**
4:05-4:30 pm O-32	**Wiley A. Hall IV**, Safe Food Alliance, Fresno, CA, USA **Integrated capillary electrophoresis and electrospray ionization (CESI) mass spectrometry for the analysis of polar pesticide residues in tree nuts and comparison with liquid chromatography**
4:30-4:55 pm O-33	**Brian Eitzer**, The Connecticut Agricultural Experimental Station, CT, USA **Collection and analysis of plant nectar and pollen and honey bee collected pollen at ornamental nurseries**

4:55-5:10 pm	**Poster Awards and Closing** **Orchid Ballroom**
6:00-10:30 pm	**Shuttle service to and from Mercato (Blue Martini)** **Outside - Hotel Entrance** *Two shuttles scheduled to run every 15 minutes*

Thursday, July 27, 2017
User Meetings

7:30-9:30 am	**SCIEX User Meeting**	**Royal Palm 1**
10:30 am-12:30 pm	**Agilent Technologies User Meeting**	**Royal Palm 3**

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NORTH AMERICAN
CHEMICAL RESIDUE WORKSHOP

Oral and Poster Presenters

(alphabetical order Last Name, First Name)

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P-3   **Examining Bioaccumulation of Select Contaminants of Emerging Concern in Clams, Mussels and Oysters**
S. Rebekah Burket, et al.; Baylor University, Department of Environmental Science, Waco, TX, USA

P-4   **Combining headspace solid phase microextraction and surface enhanced Raman scattering to detect the pesticide fonofos in apple juice**
Haoxin Chen, et al.; University of Massachusetts, Amherst, MA, USA

P-5   **Legacy Contaminant Fate in the Gulf of Mexico**
Jessica N. Landry and Kevin L. Armbrust, Louisiana State University, Baton Rouge, LA, USA

P-6   **Organophosphate Mediated Inhibition of Human Acetylcholinesterase Requires Tryptophan (Trp86) On Its Active Site: An In-Silico Analysis**
Anuj Ranjan, et al.; Amity Institute of Environmental Toxicology, Safety and Management, Noida, India

P-7   **Quantification of Toxic Metals and Antioxidants in Hot Pepper, *Capsicum* spp.**
Yogendra R. Upadhyaya et al.; Kentucky State University, Frankfort, KY, USA

P-8   **Analysis of Veterinary Drug Residues in Imported and Domestic Crawfish using Liquid Chromatography Time-Of Flight Mass Spectrometry**
Emily Wall, and Kevin L. Armbrust, Louisiana State University, Baton Rouge, LA, USA

P-9   **High-Throughput Determination of Neonicotinoid Insecticides in Pollen and Nectar using Liquid Chromatography with Tandem Mass Spectrometry Detection.**
Joe Warnick, EPL BioAnalytical, Niantic , IL, USA

P-10   **Rapid Screening and Determination of Gamma-hydroxybutyric Acid (GHB) in Dietary Supplements and Energy Drinks by GC-MS-MS**
Jason Tang, et al.; Analytical Chemistry Laboratory, NSF International, Ann Arbor, MI, USA

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Mark Krzeszowiec, NOW Foods,Bloomingdale, IL, USA

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William Meeks, Florida Department of Agriculture and Consumer Services, Tallahassee, FL, USA

P-14   **Comparison between HILIC and RP HPLC column methods for glyphosate analysis with tandem mass spectrometry**
Eugene Chang, et al.; Food and Drug Administration, Irvine, CA, USA

P-15   **Quantitative analysis of colistin in egg, milk and animal tissues by LC-MS/MS**
Guan-Jhih Peng, et al.; Taiwan Food and Drug Administration, Taipei City, Taiwan

P-16   **Development and Validation of a Method for Glyphosate and AMPA Analysis in Various Food Commodities Using Derivatization and LC-MS/MS**
John P. Zulkoski, et al.; Covance Food Solutions, Madison, WI, USA

P-17   **Residue analysis of Glyphosate, Glufosinate and metabolites using anion exchange clean-up, TMOA derivatization and LC/MS/MS**
Kathryn Hunter and Michael Conway, OMIC USA Inc, Portland, OR, USA

P-18   **Development of LC-MS/MS method for analyzing glyphosate in agricultural streams in Ohio**
Saptashati Biswas and Ellen Ewing, Heidelberg University, Tiffin, OH, USA

P-19   **Determination of Glyphosate Residues in Dry Botanical Matrices by Solid Phase Extraction-Liquid Chromatography-Tandem Mass Spectrometry**
Katarzyna Banaszewski and Vishal Patel, NOW Foods,Bloomingdale, IL, USA

P-20   **A Simplified Method for Identification and Quantification of Two Capsaicinoids in Hot Pepper Fruits**
George F. Antonious, Kentucky State University, Frankfort, KY, USA

P-21   **Validation of analytical methods for the Determination of test substances in variety of matrices**
Xianai Wu et al.; Smithers Viscient, Wareham, MA, USA

P-22   **Development of an Immunochromatographic Assay for the Detection of Feed Additive Zilpaterol**
Weilin L. Shelver and David J. Smith, USDA Agricultural Research Service, Fargo, ND, USA

P-23   **Analysis of 10 β-Agonists in pork meat using automated dispersive pipette extraction and LC-MS/MS**
Kaylee R. Mastrianni et al.; DPX Technologies, LLC,Columbia, SC, USA

P-24   **Analysis of Δ-9-tetrahydrocannabinol in plant material using automated dispersive pipette extraction and LC-MS/MS**
Kaylee R. Mastrianni et al.; DPX Technologies, LLC,Columbia, SC, USA

P-25   **The Analysis of Water for Perfluorinated Compounds using Automated Solid Phase Extraction**
Don Haertel, et al.; Horizon Technology, Inc., Salem, NH, USA

P-26   **A Novel Solution to the Analysis of Highly Complex Environmental Samples**
Scott Pugh, et al.; LECO Corporation, St. Joseph, MI, USA

P-27   **Identification and Quantitation of Pesticides in Hops Extracts with a Benchtop GC-TOFMS**
Todd Richards and Joseph E. Binkley, LECO Corporation, Saint Joseph, MI, USA

P-28   **A Flow Based Triple Quadrupole Mass Spectrometer and Its Application in Infant Formula Label Claim Analysis**
Feng Qin, et al.; PerkinElmer Health Sciences Canada, Bolton, Ontario, Canada

P-29   **Separation of 12 Natural Cannabinoids in Both Methanol and Acetonitrile Mobile Phases**
Allen Misa, et al.; Phenomenex, Inc., Torrance, CA, USA

P-30   **Complimentary Column Chemistries for Determination Pesticides in Cannabis by LC-MS/MS**
Scott Krepich and Jeff Layne, Phenomenex, Inc., Torrance, CA, USA

P-31   **Analysis of Sensory-Active Volatile Phenols in Smoke-Exposed Grapes by Gas Chromatography**
Scott Krepich et al.; Phenomenex, Inc., Torrance, CA, USA

P-32   **Separation of 12 Natural Cannabinoids in Both Methanol and Acetonitrile Mobile Phases**
Scott Krepich et al.; Phenomenex, Inc., Torrance, CA, USA

P-33   **Evaluation of the AutoMate-Q40 for Organochlorine Pesticides in Water**
Tyler Trent and Tom Hartlein, Teledyne Tekmar, Mason, OH, USA

P-34   **Determination of pesticides and persistent organic pollutants in honey by accelerated solvent extraction and GC-MS/MS**
Aaron Kettle, et al.; Thermo Fisher Scientific, Sunnyvale, CA, USA

P-35   **Multi-residue Pesticide Screening in Cereals using GC-Orbitrap Mass Spectrometry**
Paul Silcock, et al.; Thermo Fisher Scientific, Runcorn, United Kingdom

P-36   **Fast routine analysis of polar ionic pesticides in food samples by suppressed ion chromatography and mass spectrometry**
Ed George, et al.; Thermo Fisher Scientific, San Jose, CA, USA

P-37   **A Method for the Quantification of Un-derivatized Amino Acids in Sugar Snaps by LC-MS/MS**
Susanne Sales and Michal Godula, Thermo Fisher Scientific, Dreieich, Germany *(Ed George presenting)*

P-38   **Routine Quantitative Method of Analysis for Pesticides Using GC-Orbitrap Mass Spectrometry in Accordance with SANTE/11945/2015 Guidance**
Paul Silcock, et al.; Thermo Fisher Scientific, Runcorn, United Kingdom

P-39   **Robust LC-MS Analysis of Pesticides with 1.0 mm ID columns using state of the art UHPLC instrumentation**
Khalil Divan, et al.; Thermo Fisher Scientific; Hempstead, United Kingdom

P-40   **Enhancing analytical confidence and detection limits for IC and LC applications by coupling them to single quadrupole mass spectrometry**
Alexander N. Semyonov, et al.; Thermo Fisher Scientific, Austin, TX, USA

P-41   **Determination of Persistent Organic Pollutants in Fish Tissues by Accelerated Solvent Extraction and GC-MS/MS**
Aaron Kettle, et al.; Thermo Fisher Scientific, Sunnyvale, CA, USA

P-42   **Benefits of a novel automated SPME technology for the detection of environmental pollutants at trace level in water**
Evaldo De Armas et al.; Thermo Fisher Scientific, West Palm Beach, FL, USA

P-43   **Fast routine analysis of polar ionic pesticides in water by suppressed ion chromatography and mass spectrometry**
Ed George, et al.; Thermo Fisher Scientific, San Jose, CA, USA

P-44   **Time saving sample prep for the analysis of pesticide residues in Cannabis by LC-MS/MS**
Lisa Wanders, et al.; Thomson Instrument Company; Oceanside, CA, USA

P-45   **Pesticide Transformation Products: Markers of Unauthorized Application in Organic Wine Production**
Marie Suchanová, et al.; University of Chemistry and Technology, Prague, Czech Republic

P-46   **Rapid Enzyme Hydrolysis Using a novel recombinant b-glucuronidase in β-agonists Meat Analysis**
Kamolrat Metavarayuth, et al.; University of South Carolina, Columbia, SC, USA

P-47   **Determination of Chlorophenoxy and Other Herbicides in Food Commodities by QuEChERS and Detection by Liquid Chromatography with Tandem Mass Spectrometry**
John Schmitz, et al.; Covance Food Solutions, Madison, WI, USA

P-48   **Agricultural Screening of Volatile Organic Compounds as Indicators of Infestation by Portable Gas Chromatography**
Lisa Mosser, et al.; USDA Animal Plant Health Inspection Service, Miami, FL, USA

P-49   **Determination of endogenous concentrations of nitrites and nitrates in cheese: method development and validation using ion chromatography**
Susan Genualdi, et al.; U.S. Food and Drug Administration, College Park, MD, USA

P-50   **Detection of Pesticide Residues on Fruit Surfaces Using Surface Enhanced Raman Spectroscopy**
Anne-Marie Dowgiallo and Derek Guenther, Ocean Optics, Inc., Dunedin, FL, USA

P-51   **Development of Analytical Method for Determination of Florpyrauxifen-benzyl and XDE-848 acid Using LC-MS/MS**
Jung Ah Do, et al.; Ministry of Food and Drug Safety, Cheongju, Chungcheongbuk, Republic of Korea

P-52   **MRMHR: A Novel Scan Mode for Trace Level Pesticide Analysis of Cannabis Plant Extracts using High Resolution QTOF Mass Spectrometry**
Paul C. Winkler, et al.; SCIEX,Framingham, MA, USA

P-53   **Development of Simultaneous Analytical Method for Pesticide Multiresidues in Chicken and Egg using LC-MS/MS and GC-MS/MS**
Byung Joon Kim, et al.; Seoul National University, Seoul, Korea

P-54   **Proficiency-testing scheme using true samples for Gluten detection in processed food**
Caroline Laurent, et al.; BIPEA, Paris, France

P-55   **How Heterogeneity in Treated Seed Pesticide Residues Provides Challenges for Measurement Quality Control**
Jules C Carlson, et al.; Canadian Grain Commission, Winnipeg, Canada

P-56   **Pesticides in wine: evaluation of QuEChERS method and dispersive liquid-liquid microextraction for the multiresidue determination by UHPLC-MS/MS**
Osmar D. Prestes, et al.; Federal University of Santa Maria, Santa Maria, Brazil

P-57   **Evaluation of a new automated QuEChERS sample preparation technique**
Gloria de la Garza and Amy Brown, Florida Department of Agriculture and Consumer Services, Tallahassee, FL, USA

P-58   **GC-MS/MS Survey of Pesticides in Botanicals Using a Modified QuEChERS Method**
Melanie N. Parsons and Mary B. Jones, US FDA Forensic Chemistry Center, Cincinnati, OH, USA

P-59   **Evaluation of Various Dispersive Cleanup Sorbents and Their Effect on Pesticide Recovery in Cannabis**
Joan Stevens, et al.; Agilent Technologies, Wilmington, DE, USA

P-60   **A Novel Approach to Sensitive Pesticides Analysis in Cannabis by GS/MS/MS**
Melissa Churley, et al.; Agilent Technologies, Santa Clara, CA, USA

P-61   **Analysis of Cannabis for Pesticide Residues by GC/Q-TOF**
Philip L. Wylie, et al.; Agilent Technologies, Inc., Wilmington DE, USA

P-62   **Polycyclic Aromatic Hydrocarbon (PAH) evaluation in complex food matrix using Triple Quadrupole Gas Chromatography Mass Spectrometry (GC-MS/MS)**
Diana Wong, et al.; Agilent Technologies, Inc., Wilmington, DE, USA

P-63   **Multi-Residue Pesticides Screen and Quantitation in Food Matrices Using HPLC and Newly Developed Triple Quadrupole Mass Spectrometer**
Dan-Hui Dorothy Yang, et al.; Agilent Technologies, Inc., Santa Clara, CA, USA

P-64   **Analysis of Mycotoxins in Food Matrices Using HPLC and an Innovative Triple Quadrupole Mass Spectrometer**
Theresa Sosienski, et al.; Agilent Technologies, Inc., Santa Clara, CA, USA

P-65   **Method Performance of Food and Water Samples for Glyphosate and its Metabolite by LC/MS/MS**
Jerry Zweigenbaum, et al.; Agilent Technologies, Inc., Wilmington, DE, USA

P-66   **LC/MS/MS of Glyphosate and AMPA: Comparison of Direct Analysis and FMOC Derivatization**
Jerry Zweigenbaum, et al.; Agilent Technologies, Inc., Wilmington, DE, USA

P-67 **Advantages of Reversed Sandwich Injection for Pesticide Residue Analysis**
Jessica Westland, Agilent Technologies, Inc., Wilmington, DE, USA

P-68 **Reducing Run Time and Solvent Usage with EPA Method 3640A (GPC Cleanup) Using a New Column Packing Material**
Michael Tanner, et al.; J2 Scientific, LLC, Columbia, MO, USA

P-69 **Automation of EPA Method 525.2 – Determination of Organic Compounds in Drinking Water**
Michael Tanner, et al.; J2 Scientific, LLC, Columbia, MO, USA

P-70 **Improved GC/MS Analysis of Pesticides Through Inhibition of Acid-Base Chemistry in Solvents**
Michael S. Wrigley and Daniel R. Biggerstaff, o2si Smart Solutions, Charleston, SC, USA

P-71 **Analysis of L-Theanine in tea by HPLC with post-column derivatization.**
Maria Ofitserova and Sareeta Nerkar, Pickering Laboratories Inc., Mountain View, CA, USA

P-72 **Analysis of Cannabinoids using HPLC with post-column derivatization.**
Sareeta Nerkar, Pickering Laboratories, Inc., Mountain View, CA, USA

P-73 **Integration of sample cleanup and enrichment with chromatographic analysis**
Haibin Wan, PromoChrom Technologies Ltd., Surrey, BC, Canada

P-74 **High Sensitive and High Throughput Analysis of Pesticide Residues in Botanical Ingredients using GC-MS/MS**
Riki Kitano, et al.; Shimadzu Scientific Instruments, Inc., Columbia, MD, USA

P-75 **Bitter Medicine: Pesticides in Cannabis by modified QuEChERS and GC-MS-MS**
Jeffrey H. Dahl, et al.; Shimadzu Scientific Instruments, Columbia, MD, USA

P-76 **Analysis of Cannabis & Hemp Products for Heavy Metals**
Patricia Atkins and Jeff Akers, SPEX CertiPrep, Metuchen, NJ, USA

P-77 **Analysis of 648 Pesticides in Foods by LC-MS/MS Utilizing the Raptor Biphenyl Column**
Joseph D. Konschnik, et al.; Restek Corporation, Bellefonte, PA, USA.

P-78 **Preparation, UHPLC-MS/MS Analysis, and Accurate Quantitation of Nitrofurans Metabolites, Chloramphenicol, and Florfenicol in Seafood**
Fadi Aldeek, et al.; Florida Department of Agriculture and Consumer Services, Tallahassee, FL, USA

P-79 **Seafood Safety: Preparation of Seafood Samples for Dyes Analysis**
Huanwu Qi, et al.; Florida Department of Agriculture and Consumer Services, Tallahassee, FL, USA

P-80 **Stone Crabs; Heavy Metal Contamination and Risk Assessment**
Marc E. Engel, Florida Department of Agriculture and Consumer Services, Food Laboratories, Tallahassee, FL, USA

P-81 **Strategies for Extraction and Purification of Tetrodotoxin and Saxitoxin from Fish Filets with LC-MRM-MS Analysis**
Sara C. McGrath and Jonathan R. Deeds, FDA Center for Food Safety and Applied Nutrition, College Park, MD, USA

P-82 **Triphenylmethane Dye Residue Analysis in a Variety of Aquacultured Seafood Matrices including Highly Processed Canned Foods**
Wendy Andersen, et al.; Animal Drugs Research Center Denver, CO, USA

P-83 **Pesticide Multi-Residue Analysis in Red Swamp Crayfish Using QuEChERS and HPLC-MS/MS**
Yelena Sapozhnikova, et al.; USDA Agricultural Research Service, Wyndmoor, PA, USA

P-84 **Use of Chemometrics to Identify Marker Compounds**
Zhengfang Wang, University of Maryland, College Park, MD, USA

P-85 **Florida's Pesticide Residue Regulatory Program – FY 16 - 17**
Jo Marie Cook, Florida Department of Agriculture and Consumer Services, Tallahassee, FL, USA

P-86 **Ensuring your pet's health safety: Quantification of illegal, multi-class antibiotics in pet food and treats by LC-MS/MS**
Cheryl Stephenson, et al.; Eurofins Central Analytical Laboratories, New Orleans, LA, USA

P-87 **Analysis of Peptide Antibiotics in Milk using High Resolution Mass Spectrometry**
I-Lin Wu, et al.; U.S. Food & Drug Administration, Denver, CO, USA

P-89 **Evaluation of sample preparation techniques for the extraction of multiclass veterinary drugs residues in Salmon (salmo salar) by UHPLC-MS/MS**
Osmar D. Prestes, et al.; Federal University of Santa Maria, Santa Maria, Brazil

P-90 **Quantitative Analysis of Glyphosate in Regular and Infant Grain-Based Foods via LC-MS/MS Using an Anion Exchange HPLC Column**
Hugh Cramer, et al.; MilliporeSigma, Bellefonte, PA, USA

P-91 **Development of Automated Sample Preparation Method for Pesticide Analysis in Baby Foods Using Solid Phase Microextraction with Analysis by GC-MS**
Katherine K. Stenerson, et al.; MilliporeSigma, Bellefonte, PA, USA

P-92 **Analysis of Pesticide Residues in Green Tea Using QuEChERS Extraction and Cleanup with a New Dual Layer SPE Cartridge**
Katherine K. Stenerson, et al.; MilliporeSigma, Bellefonte, PA, USA

P-93 **Reducing Matrix Effects in LC-MS/MS Analysis using a Fully Automated Column Switching System**
Joerg Plagge, et al.; SCIEX, Darmstadt, Germany

P-94 **MRMHR: A Novel Scan Mode for Trace Level Pesticide Analysis of Cannabis Plant Extracts using High Resolution QTOF Mass Spectrometry**
Paul C. Winkler, et al.; SCIEX, Framingham, MA, USA.

P-95 **The Advantages of Using MS/MS ALL with SWATH® Acquisition for Confident Compound Identification in Non Target Screening (NTS) Analyses**
Paul Winkler and André Schreiber, SCIEX, Golden, CO, USA

P-96 **Application of a High Resolution Product Ion Library to Screen for PFASs**
Simon Roberts, et al.; SCIEX, Golden, CO, USA

P-97 **Determination of Microcystins In Aqueous Samples By Liquid Chromatography-Tandem Mass Spectrometry**
Craig Butt, et al.; SCIEX, Framingham, MA, USA

P-98 **SFC-HRMS a novel analytical technique for cannabinoids analysis in hemp oil**
Vojtech Hrbek, et al.; University of Chemistry and Technology, Prague, Czech Republic,

P-99 **Simultaneous determination of perfluoroalkyl substances (PFASs) and organophosphorus flame retardants (OPFRs) in dust**
Jana Pulkrabova, et al.; University of Chemistry and Technology, Prague, Czech Republic

P-100 **Streamlined Method for EPA Method 1694: Pharmaceuticals and Personal Care Products in Water**
Brian Kinsella, UCT Inc, Bristol, PA, USA

P-101   **Determination of β-agonists, including ractopamine, in animal tissues and urine by liquid chromatography-tandem quadrupole mass spectrometry**
Simon Hird and James Jowett, Waters Corporation, Wilmslow, UK

P-102   **Rapid Pass-Through Cleanup of Bovine Liver Samples Prior to UPLC-MS/MS Multiresidue Veterinary Drugs Analysis**
Kim Tran and Michael S. Young, Waters Corporation, Milford, MA, USA

P-103   **A New Strategy for the Determination of Captan and Folpet in Food Matrices**
Kari Organtini, et al.; Waters Corporation, Milford, MA, USA

P-104   **Multiresidue Pesticide Analysis in Fruit and Vegetable Commodities Using Both UPLC and APGC on a Single Mass Spectrometer Platform**
Kari Organtini, et al.; Waters Corporation, Milford, MA, USA

P-105   **Evaluation of Novel Dispersive SPE and Pass-Through SPE Options for Cleanup of High Chlorophyll Samples after QuEChERS extraction**
Michael S. Young, Waters Corporation, Milford, MA, USA

P-106   **Direct analysis of glyphosate and similar polar pesticides in various food matrices**
Dimple Shah, et al.; Waters Corporation, Milford, MA, USA

P-107   **A Novel Data-Independent Acquisition Strategy for Non Targeted, Accurate Mass Contaminant Screening**
Gareth Cleland, et al.; Waters Corporation, Milford, MA, USA

P-108   **The analysis of natural and synthetic estrogens at low ppq levels in surface water and final effluent water by liquid chromatography-tandem quadrupole mass spectrometry**
Simon Hird, et al.; Waters Corporation, Wilmslow, UK

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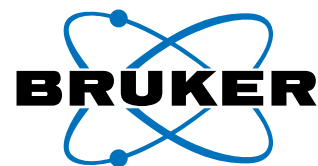
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ORAL PRESENTATION ABSTRACTS

A-1   New Tools for the Determination of Chemical Constituents in Complex Matrices

Kevin A. Schug,[1] Harold M. McNair[2]

[1]Department of Chemistry & Biochemistry, **The University of Texas at Arlington**, Arlington TX
[2]Department of Chemistry, Virginia Tech, Blacksburg VA

Close collaboration with industry partners has afforded opportunities to assist in the development and application of new tools for chemical measurement. Such undertakings are characterized by a mix of fundamental aspects of sample preparation, chromatography, and detection with practical aspects in the application of the technologies to solve problems. Two tools that have been a major focus of research in the Schug lab are a) a vacuum ultraviolet spectroscopic detector for gas chromatography (GC-VUV) developed by VUV Analytics, Inc., and b) an on-line supercritical fluid extraction – supercritical fluid chromatography – mass spectrometry (SFE-SFC-MS) developed by Shimadzu Scientific Instruments, Inc. GC-VUV has been applied in a variety of different areas, including gasoline characterization, fatty acid profiling, and the discrimination of isomeric environmental contaminants. Means by which GC-VUV can offer a complementary solution to GC-mass spectrometry, especially for distinguishing isomers, will be discussed in the context of recent applications. SFE-SFC-MS represents a new and broadly applicable solution for the combination of streamlined sample preparation with high efficiency separations and specific detection. Applications explored to date include the determination of drugs of abuse in urine, polyaromatic hydrocarbons and their metabolites in soil, and fatty vitamins in tablets. The multitude of variables for optimization of the system make it flexible and powerful; however, we seek to develop and disseminate some practical guidance to help enable routine use of the system.

O-1   The use of GC-Orbitrap-MS in multi-residue analysis of pesticides

Paul Zomer, Marc Tienstra, Hans Mol

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The introduction of GC-Orbitrap-MS systems expands the application of full-scan high-resolution MS detection to gas chromatographic analysis, which is still an important and necessary addition to LC-MS based multi-residue methods for pesticides.
Compared to electrospray ionization used in LC-MS, the electron ionization generates, in most cases, a number of selective fragments for each compound, thereby eliminating the need for alternating fragmentation scan events before detection. A resolving power of 60,000 (at m/z 200) was chosen as this provides sufficient resolution to achieve good mass accuracy (<5 ppm) for the compounds down to low ppb levels in complex matrices. At this setting, approximately 5 scans/sec, (≥15 points across an average GC peak) are obtained, enough for a good quantitative performance.
The widely used (acetate buffered) QuEChERS method was used for sample extraction and clean-up. The final extract contained 0.25 g/mL, and 5 μl was injected into the GC.
From the different options to process the data, the approach using two sensitive and selective fragment ions per compound was chosen. This allows for efficient data processing and generates data that, combined with the ratio between the two ions, gives enough information to fulfill the requirements for compound identification according to the EU guidance document on pesticide residue analysis (SANTE 11945/2015).
Results of the validation of a GC-Orbitrap-MS method for cereals/feed ingredients and pre-harvest vegetation samples will be presented.

O-2   Gas Chromatography - Vacuum Ultraviolet Spectroscopy: A New Tool for Food and Environmental Analysis

Jack Cochran, Alex Hodgson, James Diekmann III, Lindsey Shear-Laude, Sean Jameson, Dale Harrison

VUV Analytics, 715 Discovery Blvd, Ste 502, Cedar Park, TX 78613, USA; jack.cochran@vuvanalytics.com

A vacuum ultraviolet (VUV) detector has been developed for gas chromatography that collects spectral data from 120 to 430 nm. Almost all chemical compounds absorb light in the VUV range, so the detector can be considered universal. VUV absorbance spectra are unique, even to the point of allowing isomer differentiation. Having unique spectra also

supports deconvolution of coeluting peaks in gas chromatography and makes chromatographic compression possible, i.e., the speed-up of run times that allows higher throughput. The detectability for compounds for the VUV detector ranges from low to mid pg, depending on the compound. Interestingly, compounds like formaldehyde and water can be detected, which are typically hard to determine with other detection means. Spectral filters applied post-data collection through software selection allow for compound class selection, which is helpful in distinguishing target compounds from matrix during data review and quantification. Relevant applications include formaldehyde-in-food, Mineral Oil Saturated Hydrocarbons/ Mineral Oil Aromatic Hydrocarbons (MOSH/MOAH) in food packaging, and Total Petroleum Hydrocarbon analysis for environmental samples.

O-3   LC-HRMS, Mass Spectral Libraries, and Compound Databases for Screening Chemical Residues and Contaminants

Jon W. Wong[1], Kai Zhang[1], Douglas G. Hayward[1], Kelli Simon[1,7], James B. Wittenberg[1], Hoon Yong Park[2,8], Jian Wang[3], Chia-Ding Liao[1,4], Zhengwei Jia[5,9], and James S. Chang[6]

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[6] Thermo Scientific, San Jose, CA, USA
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[8] Current Address: BD Diagnostic Systems, Hunt Valley MD
[9] Current Address: Waters Corp., Shanghai, People's Republic of China

Liquid chromatography-high resolution mass spectrometry (LC-HRMS) can be utilized for screening chemical residues and contaminants, such as pesticides, mycotoxins, and veterinary drugs, in complex food matrices. Quantitation and identification of these residues and contaminants require the selection of characteristic precursor and product ions. In this work, mass spectra libraries for over 1000 chemical contaminants were compiled from experimental data obtained using a LC-Quadrupole-Orbitrap MS operated in positive electrospray ionization mode with full scan and data-dependent MS/MS acquisition. The assembly of HRMS libraries supports the assignment of fragmentation for chemical contaminants acquired in previous low resolution MS/MS studies. These libraries can be utilized to inform the selection of product ion transitions and the development of a compound database for targeted LC-MS/MS methods using a triple quadrupole instrument. The application of compound databases and software algorithms will be presented to demonstrate the identification of chemical residues and contaminants in foods by LC-HRMS. The combination of an LC-HRMS screening method with generic sample preparation procedures (e.g. QuEChERS and dilute-and-shoot) can ultimately be used to screen a large number of compounds of interest in a variety of foods.

O-4   Approaches for the (broad) screening of veterinary drugs by full scan accurate mass determination

Eric Verdon, Dominique Pessel, Sophie Mompelat, Murielle Gaugain, Estelle Dubreil, Pierrick Couedor

ANSES-Laboratory of Fougeres, European Union and National Reference Laboratory for Veterinary Drug Residue Control in Food from Animal Origin, BIOAGROPOLIS, 10B rue Claude Bourgelat, Parc d'Activite de la Grande Marche , Javene, CS 40608 F-35306 Fougeres, France ; eric.verdon@anses.fr

For quite a long time now the current analytical methods dedicated to 1 - a large scope of screening and 2 - to accurate confirmation of veterinary chemical residues in food are mainly based on use of tandem mass spectrometers coupled to liquid chromatographic separation techniques, the so-called LC-MS/MS instruments. Over the past decade, advances in MS detectors have opened brand-new smart routes in analytical strategies to monitor chemicals at residue level in food by means of high resolution MS detectors such as TOF and Orbitrap devices. Such LC-HRMS instruments capable of combining a Full Scan mode detection of molecular ions together with an accurate High Resolution mass measurement are perfectly fit for the screening in food extracts and in a one shot injection for a large number of chemicals at high speed and at already quite sensitive detection levels. Meaning that these residual substances from veterinary treatments need no longer to be monitored on a targeted basis by using suitable methods for each families or classes of substances (such as antimicrobials, antiparasitics, anti-inflammatories, tranquilizers, …). New strategies for screening are now opened either to a post-targeted analysis with embarked libraries or even to a non-targeted approach with external data-mining for molecular identification. This concern will be displayed and illustrated throughout the presentation focusing

on the antimicrobial veterinary residue control in food. A particular interest will be set on the context of validating such a broad-spectrum screening and the criteria of performance that could be requested to give confidence in reliable-enough methods for their accreditation.

O-5 LC-MS/MS Methods for an Effective Control of Veterinary Drugs in Raw Materials and Manufactured Products – A Food Industry Perspective

Thomas Bessaire, Adrienne Tarres, Andrea Beck Henzelin, Claudia Mujahid, Marie-Claude Savoy Perroud, Lucie Racault, Pascal Mottier, Thierry Delatour and Aurélien Desmarchelier.

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In modern agricultural practices, more than 400 veterinary drugs are used to treat or prevent infection, and to improve weight gain and feed efficiency. Their incorrect use in animal production as well as the disrespect of withdrawal time after treatment may lead to residues in various edible animal tissues. Since such residues may have toxic effects on consumers, organizations such as Codex Alimentarius, European Union or the US Food & Drug Administration have established Maximum Residue Limits (MRL) for veterinary medicinal products in foodstuffs from animal origin or have totally banned some of them. Therefore, the need to have methods capable to screen and confirm the possible presence of a broad range of these substances in food is of utmost importance with the ultimate goal to protect consumers.
In that regard, a set of four LC-MS/MS methods is presented for screening 151 drug residues in raw materials, processed ingredients and finished products based on milk, meat, fish, fat and egg. A first multi-class, multi-residue method covers 105 analytes including families of amphenicols, macrolides, quinolones, sulfonamides, avermectins and coccidiostats. Due to analytical constraints, specific family methods have been developed for the determination of 10 tetracylines, 23 beta-lactams and 13 aminoglycosides, respectively.
The presentation will illustrate the journey of an analytical method as done in the industry, from the initial development and validation phases to their deployment in different routine Nestlé laboratories.

O-6 RapidFire Mass Spectrometry with Enhanced Throughput as an Alternative to Liquid-Liquid Salt Assisted Extraction and LC/MS Analysis for Sulfonamides in Honey

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The use of veterinary drugs in honey bees for the prevention of infectious disease is ever increasing due to the spread of colony collapse disorder around the world. The United States Food and Drug Administration is concerned about the presence of these drugs residues in honey as they often lead to health concerns or potential antibiotic resistance. Currently there is a need for a rapid screening method for the detection of veterinary drugs in honey. Herein is a method that utilizes automated solid-phase extraction that is directly coupled to a mass spectrometer for the quantitative screening of 12 different regulated sulfonamides in honey. Identification of the residues was performed using MS/MS with a triple quadrupole mass spectrometer. The acquisition method developed for this analysis can extract with the automated solid-phase extraction system and analyze a single sample on the mass spectrometer in approximately 20 seconds, with minimal sample preparation. A target testing limit of 10 ng/g in honey for the sulfonamides was used based upon action limits set for other food commodities regulated by the United States Food and Drug Administration. A complete method validation procedure was conducted to evaluate the effectiveness of this quantitative screening method.

O-7 Quantitative analysis with Orbitrap™ high-resolution LC/MS/MS and a new triple quadrupole LC/MS/MS for validation of a multi-residue veterinary medicine suite in cattle muscle

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Due to higher production demands of dairy and meat products, different classes of veterinary drugs are used (often illegally at higher than the permitted doses), in both animal feedstuff as well as directly into the animals. This can result in high level residues of these compounds being found in food products being available commercially for human consumption.

The aim of this work was to develop methods on two MS platforms for 167 veterinary products , with 56 labeled internal standards, from the following classes of veterinary medicines: Cefalosporins, macrolides, penicillins, quinolones, sulfas, tetracyclines, anthelmintics, nitroimidazoles, NSAIDs, sedatives, avermectins and coccidiostats. The work was based on guidelines from EU 2002/657/EC. HRAM methods were developed on a Thermo Scientific QExactive LCMS High Resolution Mass Spectrometer coupled to a Thermo Scientific Ultimate 3000 RSLC system. Full scan data dependent MS/MS as well as data independent acquisition (DIA) modes were used. In addition, a cloud based HRAM library (mzCloud) was built to help with the identification of suspected contaminants (target screening).   At the same time, a triple quadrupole MS was used in the study to evaluate the quantitative comparison of HRAM and traditional tandem MS used today in most food testing laboratories.

A QuEChERs-like sample preparation method was optimized to reduce matrix co-extractives. Results for cattle muscle show excellent quantitative performance in both MS platforms, with HRAM providing the opportunity for target screening with advanced processing software.

O-8   Determination of the Bis(2-ethylhexyl) phthalate (DEHP) Concentration of Beer Stored in Bottles with PVC Gaskets

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PVC is a common food contact material that is usually plasticized to increase its flexibility. Phthalates are one of many chemical compounds that are used as plasticizers in PVC.   They may be used in packaging materials for foods and can also be found in components of certain food processing equipment such as conveyor belts and tubing and/or hoses. Transfer of phthalates from packaging to the surfaces of foods can occur. In recent years, there has been interest in understanding the health effects of phthalates, as well as the possible human exposure levels. However, there is limited information available about the major routes of exposure to phthalates. A recent study investigated the plasticizers currently used in PVC food packaging and processing materials. It was determined that most products no longer contain DEHP as the primary plasticizer. One of the main product types where it was still found was in the PVC gaskets of beer bottle lids. In the current study the concentration of DEHP that had migrated from the gasket into the beer was investigated. In order to minimize sample preparation and blank issues while maintaining a low detection limit for DEHP, a stir bar sorptive extraction (SBSE) coupled with GC-MS/MS was used to extract and quantify the DEHP in the samples. To ensure that the DEHP was coming from the packaging material and not from other food contact surfaces encountered during the fermentation and bottling steps, beers packaged in both bottles with and without PVC gaskets were investigated.

O-9   LC/MS/MS Determination of Per- and Poly-Fluoroalkyl Substances (PFAS) in Eggs and Dairy Products

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The ubiquity of per- and polyfluoroalkyl substances (PFAS) means that they can be found in nearly every corner of globe, which is in no small part due to the persistent nature of these unique compounds. PFASs are man-made compounds that are found in a myriad of products such as fire-fighting foams, food packaging, and stain-resistant household items. These present a major contribution to the environmental presence of PFASs—industrial manufacturing byproducts, landfill leachates, and deliberate discharge from fire-fighting activities tend to contaminate ground and surface water sources. Studies have also shown that at elevated levels, the consumption of PFASs could lead to negative health effects in certain individuals. This lead the USEPA to establish a lifetime exposure health advisory limit of 70 ng/L in drinking water for two PFASs: PFOA and PFOS.

The unique chemical properties of PFASs render them persistent and highly bioaccumulative—raising the question of accumulation in livestock and whether they can be passes along to food products such as eggs and dairy products. To this end, the work presented here describes a robust and sensitive method for the determination of twenty-three PFASs including short- and long-chain perfluorocarboxylates and perfluorosulfonates, sulfonamides, and fluorotelomer alcohols using UHPLC-MS/MS coupled with QuEChERS cleanup. By leveraging the extreme sensitivity of modern commercial mass spectrometers and aggressive sample cleanup procedures, this technique can reach parts-per-trillion reporting limits for PFASs even in complicated matrices such as eggs and cheese.

O-10   A Novel Method for Analysis of 3-monochloropropane-1,2-diol (3-MCPD), 2-monochloropropane-1,3-diol (2-MCPD) and Glycidyl Esters in Oils and Fatty Foods Using GC-MS/MS

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An emerging concern in recent times is the non-genotoxic carcinogens, 3-monochloropropane-1,2-diol (3-MCPD), 2-monochloropropane-1,3-diol (2-MCPD) and gycidyl esters, found in a variety of edible oils and food matrices including infant formula. These process contaminants are formed in the manufacturing process of foods containing vegetale protein and can also be produced by heat processing in the presence of fat. Risk assessments have been performed for these analytes and MCPD has been deemed to be a major contributor for health concerns like kidney tumors or renal tubular hyperplasia. Due to strict EU regulations for MCPDs in food and oil products, international companies are pouring efforts in to minimize the presence of MCPDs in oil and food products. The health hazards of MCPDs necessitate the need to develop a robust and sensitive methodology that can accurately detect MCPDs in food products. Eurofins CAL have been in the forefront in establishing a method that is robust and unique. In the current method, fats are extracted from food matrices, such as infant formula, using a novel and distinctive fat extraction procedure. The next step involves alkaline catalysis of extracted fats and oils to cleave the esters followed by derivatization of diols with phenylboronic acid and extracts are analyzed using GC-MS/MS. Linearity studies and method validations were performed in several matrices, and the resulting protocol is determined to be fit for the intended use.

O-11   A one-year experience with pesticide residue analysis in hops using QuEChERS based method: Living up to expectations or blind alley?

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In 2016 we introduced a method for the analysis of pesticide residues from hops using sample preparation procedure based on QuEChERS approach in combination with dispersive solid phase extraction (dSPE) cleanup. The validation is now presented of this method for analysis of 69 pesticides in hops matrix, using LC-HRMS/MS positive ESI mode. A critical part of the method, the dSPE cleanup, was thoroughly optimized to achieve maximal mitigation of the strong matrix effects causing massive signal suppression and thus difficult quantification. The sorbent blend including primary secondary amine (PSA), C18 and zirconia-based sorbent was finally used for method validation. The method was successfully validated in terms of recovery and reproducibility by spiking hops at two levels 50 and 500 μg/kg, six replicates per level.
The method was applied for monitoring pesticide residues in hops samples coming from the 2016 harvest in Czech Republic. The amount of pesticide residues in all analyzed samples passed the MRL criteria established by EU regulation. Hand in hand with the analysis of hops as key ingredient, the pesticide residues in beer should also be monitored. Therefore, in our set of experiments we focused on pesticide residue analysis in various beer samples. Our attention focused on modification of clean-up strategy of QuEChERS extract to achieve as low detection limit as possible. We probed currently popular crafted beers for pesticide residues and the resulting data from monitoring pesticide residues in beer were compared to other food matrices to demonstrate their relevance.

O-12   Incurred Residues-What Are They and How Much Is Present in Our Food Supply?

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Incurred pesticide residues are residues that are formed after treatment under *in vivo* conditions such as crops under agronomic conditions or livestock under animal husbandry conditions. Formation of incurred residues generally cannot be duplicated in a laboratory setting as they are based on biological and environmental inputs. Several mechanisms may be involved in the generation of incurred residues. These include chemical modification of the pesticide to form conjugates or other metabolites, chemical binding to various plant or animal components, and non-specific physical entrapment. Plant and animal metabolism studies required by regulatory authorities worldwide are designed to identify these various residues. Radiolabeled materials are employed so material balance can be monitored and the route of degradation can be determined. Based on these metabolism studies the total toxic residue (TTR) is established. The TTR

is composed of the parent pesticide and metabolites of toxicological concern. Analytical methods are then developed to measure all components of the TTR which are ultimately used for tolerance enforcement. The tolerance enforcement methods must be validated by analyzing actual metabolism samples and showing concurrence with the metabolism study results (radiovalidation). The method must account for ≥ 70% of the TTR in the aged samples, as identified in the metabolism study. The resulting enforcement method thus accounts for incurred residues if present. This presentation will discuss the importance of metabolism studies for determining incurred residues, leading to subsequent tolerance enforcement method development and radiovalidation. The importance of these methods as they relate to pesticide screening for food safety will be discussed.

O-13   How low can we go? Study to assess the accuracy of different test sample weights in pesticide residue analysis of fruits and vegetables using different chopping methods

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Standard sampling protocols from regulatory agencies, monitoring programs, and field trial applications in pesticide analysis typically call for comminution of at least 1 kg collected samples prior to subsampling and analysis of test portions. In part due to lack of sensitivity of analytical instruments, FDA methods originally called for 100 g test portions. Reducing subsample sizes has a direct and substantial effect to streamline methods, lower costs, and improve laboratory efficiency. As technologies and techniques advanced over time, common test portion sizes decreased to 25-50 g and then 10-15 g currently. Many studies have been conducted that investigate different matrices, analytes, and comminution tools/methods to minimize test portion size while still maintaining representative results for the original bulk sample collected. Cryogenic comminution with dry ice or liquid nitrogen have been shown to reduce degradation and volatilization of certain susceptible pesticides, but this is inconvenient in routine practice and care must be taken to avoid condensation of water from the atmosphere getting into the sample. In this study, the Blixer chopper at room temperature was compared with the Cryomill using liquid nitrogen for the analysis of many incurred and spiked pesticides in 10 commodities (apple, banana, broccoli, celery, grape, green bean, orange, peach, potato, squash). Although 75-500 mg test portions have been shown to be acceptable in terms of accuracy for certain pesticides and matrices in other studies using the Cryomill, a 96-well plate format and careful logistics are needed when using such small portions. We compared 15, 10, 5, 2, and 1 g for the Blixer with 5, 2, and 1 g Cryomill subsamples in this study using manual batch sample preparation followed by UHPLC-MS/MS and automated sample mini-cartridge SPE cleanup and low-pressure GC-MS/MS. The results showed that 1-5 g portions gave similar accuracies using the Cryomill (of 20 g Blixer subsamples), but 1-2 g Blixer subsamples were often less accurate than 10-15 g. For most pesticides/matrices, 5 g test portions gave acceptable trueness with precision of sample preparation <10% RSD, but some notable cases occurred in which larger test portions were still warranted. The uncertainty contribution of sample processing was calculated and plotted vs. subsample size in each approach, and the chemist can make informed decisions about which sample test portion size to use based on the degree of accuracy desired in the analytical application.

O-14   Advantages of Cryogenic Milling using Liquid Nitrogen for the Routine Analysis of Pesticide Residues in Food and Feed

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Determining pesticide residues in food and feed within the scope of single-residue (SRM) or multi-residue methods (MRM) an accurate comminution is a key prerequisite for analysis validity. Cryogenic milling using dry ice is still considered to be generally accepted for ensuring pesticide stability during homogenization procedure. Yet, it is required to first blend and subsequently mill the samples. Remarkably liquid nitrogen (LN_2) as a superior alternative cooling agent has hardly been used for pesticide residue analysis. A successfully applied cryogenic comminution technique using LN_2 and allowing high-throughput sample homogenization is presented. Complying with current quality control requirements, various food and feed commodities with different sample sizes can be comminuted in the presence of LN_2 using standard equipment and following the safety requirements. Not only do samples not need to be blended and frozen prior to comminution, but also loss of labile or volatile analytes is avoided due to the very low temperatures (-196 °C). As a result, the full performance of laboratory mills can be utilized. Further advantages are no condensation of air moisture or any changes in sample weight and no interruption of sample processing after milling. Additionally, a method

modification implementing LN_2 for cryogenic comminution of the entire sample by determination of dithiocarbamates as CS_2 shows a significant increasing of Thiram recoveries up to 95 %, which has been successfully proven by a proficiency test. Conclusively, the use of LN_2 allows full implementation in the daily routine, prevents losses of pesticides and increases analytical quality.

O-15   Utilization of 96 Well Plates in Residue Analysis. Micro-Extraction is a Fast, Cost Effective, and Green Technology to Determine Residue in Various Matrices

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The 96 well plates are routinely utilized by various disciplines in the chemical industry, and yet they were not widely incorporated in residue analysis in agricultural labs. A sample throughput of 96 samples/day allows for a fast turnaround on sample analysis (especially in emergency situations) and is very friendly to the environment. The challenge to incorporate 96 well plates into residue analysis was to effectively represent an agricultural sample while reducing the size of the sample to fit into a well plate.
Close to 20 years ago, BASF began investigating reducing sample size in residue analysis to fit the 96 well plates. To achieve the smaller sample size, the "Micro-Extraction" residue analysis technique and automation were developed at BASF, RTP, NC.
This technology was used to analyze thousands of samples for many AIs over the years that covered over 100 different matrices (various crop, soil, water types). The "Micro-Extraction" technology was utilized by Crop Life International to help screening for over 90 AIs for recycling the plastic containers.
This presentation will focus on many of the aspects of the utilization of 96 well plates and illustrate how we resolved the hurdles we faced: homogenization, wet chemistry, different matrices, and proving to regulatory agencies across the globe that this technology can be rugged, reproducible, reliable, and friendly to environment.

O-16   Isotopically labelled compounds, nuclear and isotopic techniques in food safety monitoring

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Tandem mass spectroscopy coupled with liquid and gas chromatography has simplified and enhanced analytical methodology in food safety monitoring. However, the advanced technology while enabling reduced clean-up procedures has led to data reliability problems due to matrix effects and reduced / enhanced recoveries. Compensating for these effects and improving data reliability has been a problem and is also not favourably considered by some national regulatory agencies. The use of stable isotope (SI) internal standards (IS) is a natural fit to compensating for matrix effects and instrumental issues resulting in improved reliability of results. SI labelled standards are not cheap or easy to obtain although they are becoming more readily available these days. Another application of isotopic techniques in food safety is the use of radio-labelled pesticides to measure recoveries of incurred residues, a problem not previously addressed by QuEChERS type methods. Techniques for the use of SI IS as well as radio labelled compounds in food safety monitoring, when best used and, maximizing result reliability and analysis speed while simultaneously reducing costs will be discussed.

O-17   Chemical & Hop Laboratory – 65 years of Accomplishments and Continuing Challenges

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For the past 65 years the Chemical and Hop Laboratory has served to support several agency programs; analyzing samples for pesticide compliance, commercial feed and fertilizer for label guarantees, screening food for chemical residues, grading and testing hops for quality, and participating in USDA's Pesticide Data Program. In order to meet client needs the lab regularly restructures, resulting in a dynamic organization that is constantly changing. Currently this lab is organized into sections based on sample type: Food, Feed, Fertilizer, Environmental, Hop Inspection, and Hop Chemistry.

Each section is staffed and managed separately (with some crossover), but is overseen by the same administrative and quality assurance units. Throughout the years this laboratory has evolved to meet the changing technologies and testing requirements: transitioning from wet chemistry techniques to non-selective detection with and without chromatography in 1993, updating to selective based detection systems in 2003, and gaining ISO 17025 accreditation in 2008. In meeting the plea "to do more with less" this labs analytical capability has consistently increased throughout the years, mostly as a direct result of newer technologies and advances made in bench chemistry techniques. One of the most recent and major changes for this laboratory is the replacement of its outdated LIMS with a newer version that has several new features, including the ability to import results directly from instrumentation. A brief history of the many changes this laboratory has gone through, as well as some of the analytical and operational challenges it has faced will be presented.

O-18   Predicting Mercury Exposure to Women of Child-Bearing Age From Various Grouper Consumption Scenarios

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Grouper is a finfish that is readily available to and popular with both recreation and retail fish consumers. Grouper samples were collected from the Gulf of Mexico (GoM), the largest commercial grouper fishery in the USA, from 2014-2016 (n=114). Mercury concentrations in fish were determined by ICP-MS. These mercury concentrations were subjected to Monte Carlo simulation to determine the distribution of exposure levels to women of child-bearing age (WCBA), and exposures evaluated for health risk to consumers by comparing exposure to the US EPA methylmercury reference dose. Health risk was evaluated for WCBA consuming either 1 meal per week, or 2 meals per week – the USDA Dietary Guidelines for Americans recommendation.

O-19 Analysis of Polar Pesticides Using a Mixed-Phase Mode HPLC Column

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Highly polar pesticides and plant growth regulators are poorly retained on a conventional reversed-phase HPLC column; therefore, they are not included in the routine pesticide screening methods. They have vastly different molecular structures and characteristics from very basic (paraquat) to very acidic (glyphosate).   Several LC/MS methods were developed (QuPPe) by using different HPLC columns including anion-exchange, carbon, normal phase, and HILIC (hydrophilic interaction liquid chromatography) using separate mobile phase compositions. This makes the screening method a bit complicated as a chemist must change the column/mobile phase combination to screen these analytes. This talk will discuss LC/MS methods using only one mixed-phase mode column which possesses reversed-phase, anion-exchange, and cation exchange properties to screen a variety of very polar compounds. By changing different mobile phase parameters including pH, salt and acetonitrile concentration, this column can be used for many types of analytes. The methods were successfully validated to determine glyphosate, glufosinate, AMPA, paraquat/diquat, ethephon, fosetyl aluminum and maleic hydrazide in food. More analytes are being added to expand the analytical capability of this versatile column in order to simplify and expand the pesticide screening capability.

O-20   An ion pair reverse-phase liquid chromatography method coupled to tandem mass spectrometry for analysis of glyphosate and its metabolites

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An ion pair reverse-phase high performance liquid chromatographic method is developed and optimized for determination of glyphosate, N-acetyl glyphosate, N-acetyl-aminomethylphosphonic acid and aminomethylphosphonic acid (AMPA). Tetrabutylammonium formide is used as the ion-pairing reagent at pH 2.8. A triple-quadrupole tandem mass spectrometer is coupled to the liquid chromatographic system to detect the compounds in negative electro-spray ionization (ESI) mode. The stable-isotope dilution methodology in combination with LC-MS/MS in this study provides

high analytical specificity for quantitative analysis of the four compounds at 10 ng/g fortified range in foods by using the extraction methods on FDA LIB 4596. The recovery of all four compounds is within 80-120%. A HILIC chromatography method is also developed and compared with the ion pair reverse-phase chromatography method. The results show that ion pair reverse-phase chromatography is better.

O-21   Seeing the Trees through the Forest – Analytical Challenges and Strategies for Residue Analysis in Cannabis

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Legalization of medical and recreational cannabis has stimulated exponential growth in cultivars of this potent plant. Like the entirety of traditional agriculture, cannabis cultivators are challenged with pests that regularly threaten their crops' health. Insects, mold, and mildew pressures are the reason so many cannabis growers have turned to potent, active-chemical pesticides to protect and sometimes save their crops – and financial investments. However, these chemicals are prohibited in most states, establishing the need for testing.

Pesticide residue analysis of cannabis is challenging because the cannabinoids and terpenes are present in high abundance relative to the pesticides. Many techniques have been discussed over the past several years incorporating either QuEChERS or SPE. MS full scan data shows these techniques alone do not remove substantial amounts of co-extracted materials, which will lead to chromatographic challenges, inaccurate results and increased instrument downtime. PAL has developed a straightforward yet very effective sample preparation approach to analyze pesticides in cannabis resulting in ppb quantification on both LC-MS/MS and GC-MS/MS. This sample preparation approach substantially reduces matrix effects, allowing the chemist to fully utilize the sensitivity of MS/MS systems and increase instrument productivity. The scope of the study validated over 200 compounds which meet the requirements for states requiring multi-residue pesticide testing in cannabis. Data presented will show the drastic improvement in background matrix removal, precision and accuracy for both LC-MS/MS and GC-MS/MS analysis utilizing this approach.

O-22   Cannabis Pesticide Residue Testing Implementation: The Colorado Experience

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In 2012, non-medical retail cannabis became legal in Colorado for adult use. Due to the federal prohibition on cannabis, specific guidance and standards from federal agencies for ensuring cannabis safety do not exist. As an example, the Environmental Protection agency has not performed any scientific risk assessments of the potential health hazards posed by application of any pesticide to cannabis, and therefore, no pesticides have been specifically approved for use on cannabis and no maximum residue levels have been determined. As a result, the State of Colorado has determined that, until scientific assessment establishes which pesticides can be safely applied to cannabis, all cannabis contaminated by a pesticide not approved for use by the Colorado Department of Agriculture constitutes a threat to public health and safety. From an analytical perspective, this position presents two unique challenges: first, of the thousands of unapproved pesticide active ingredients, which compounds are Colorado's regulated marijuana testing facilities expected to be able to identify; second, in the absence of defined tolerance limits in cannabis, and given that any method's limit of detection will be somewhere above zero, at what level shall a pesticide be considered present considering that each laboratory method will have varying limits of detection? The State of Colorado, in conjunction with private Colorado marijuana testing facilities, has initiated three interlaboratory pesticide residue detection limit studies to find solutions to these difficult questions.

O-23   Pesticide Residues in *Cannabis*: Pesticide Exposure Risk Assessment

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The registration of pesticides for use in food and feed crops production agriculture requires the development of a dietary risk assessment. Dietary exposures based on food and water consumption are compared with known toxicological endpoints to evaluate potential risk and establish allowable residues in food crops. Residue levels from magnitude of residue studies are used to develop tolerances or MRLs. Currently, no pesticide labels for agrochemical use on *Cannabis*

have been approved under the FIFRA. Thus, legal growers have very few, if any, approved options for pesticide use during production. Pesticides generally used in commercial food and ornamental production agriculture are clearly prohibited as growers must adhere to strict label requirements, however reports in the press and literature continue to show residues of such products. Consumers are concerned about the potential health risks from these residues. Many state and private analytical laboratories are beginning to analyze for pesticides, looking for products used legally in food and garden applications. If prohibited pesticides are found these batches are removed from commerce. Until testing is required on a wider scale, there continues to be concern about the potential health risks. The EPA's Dietary Exposure Evaluation Model (DEEM) and Stochastic Human Exposure and Dose Simulation (SHEDS) are tools used by regulatory authorities to assess risk from human pesticide exposure. Using these models and data from pesticides levels that have been reported in the literature for *Cannabis*, an evaluation of exposure risk to pesticides was performed. Potential inputs from both dietary and inhalation exposures were evaluated.

O-24   The Roles of National Associations in State and Federal Regulatory Cooperation: Implications for Future Cannabis Policy

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The national associations representing state and federal regulatory entities play critical roles in nurturing partnerships between these two groups. These partnerships will be critical as the states and federal government will seek to harmonize regulatory processes that will foster interstate commerce as cannabis products become increasingly prominent in the marketplace. Many lessons can be learned from the manner in which pesticide and food safety regulations have evolved and the roles that national associations play, providing a platform for intercourse between regulatory partners as well as the regulated industry. For example the Association of American Pesticide Control Officials (AAPCO) and the State FIFRA Issues Research and Evaluation Group (SFIREG) will provide the platforms for regulatory policy discussions between EPA and the states of issues concerning pesticide use while the Association of Food and Drug Officials (AFDO) will undoubtedly play a similar role for discussions pertaining to FDA and state officials with food safety responsibilities. Regulatory issues concerning the use cannabis production byproducts in animal feed will be addressed at meetings of the Association of American Feed Control Officials (AAFCO). These associations have greatly improved the processes that exist today and will continue to play crucial roles in a future world of legal cannabis.

O-25   Challenging analytical strategies to characterize the cocktail of chemicals occurring in *Cannabis* plants and products thereof

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In the recent decade, the interest in phytochemicals occurring in Cannabis based products has been increasingly growing. In addition to control of major cannabinoids (such as THC, THCA, CBD, CBDA, CBDV, CBG, CBGA, CBN, CBC..) in various foodstuffs enriched by various cannabis extracts, characterization of an enormously complex cocktail of bioactive chemicals contained in cannabis plants has become a challenging task, specifically in the context of its medical uses. In our study, a number of mass spectrometry based analytical strategies applicable for (i) a targeted rapid cannabinoids screening, (ii) profiling of entire set of almost 300 related compounds and (iii) metabolomics fingerprinting were optimized and validated, performance characteristics were determined. To our experience, coupling the supercritical fluid chromatography with the high resolution mass spectrometry (SFC-HRMS/MS), employing ion mobility (IM) as the 3rd separation dimension, represents the most efficient analytical platform enabling an in-depth analysis of cannabis based matrices, including those with a high fat content. As regards volatile components of Cannabis metabolome (represented mainly by various terpenes), multidimensional gas chromatography coupled with mass spectrometry (GCxGC-MS) was employed for their profiling. In the final part of this presentation, special attention will be paid to a Quality Assurance /Quality Control (QA/QC) issues in cannabinoids analysis, troubleshooting experiences will be presented and discussed.

O-26   Dye residues in aquaculture products: targeted and metabolomics mass spectrometric approaches to track their abuse

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Chemotherapy has been applied in aquaculture for the last decades and is of growing concern because of current safety worries: environmental contaminants, emergence of resistance to antibiotics, consumer demand on healthy foods… Among these chemicals, several pharmacologically-active dyes like the pretty well-known malachite green (MG) may be administered because they are cheap and they bear interesting antiseptic and antibacterial activities. More interest was recently focused on the possible use of similar compounds to MG that would not be sought in aquaculture products. But treatment based on MG and other related dyes is now prohibited due to toxicity concerns. Then appropriate analytical approaches are needed to control their abuse. The aim of the study was to initiate an exhaustive strategy of control by implementing both targeted and non-targeted approaches. On the one hand, the strategy of reliable and high-throughput targeted mode was proposed considering all the dyes of interest. A LC-MS/MS method was validated to analyze 14 dyes belonging to different related families. An oxidative step was integrated in order to recover the parent forms for the dyes which are supposed to metabolize in reduced forms after administration to fish. On the other hand, the non-targeted approach was conducted to investigate the potential presence of biomarkers after treatment of farmed fish and selecting for this research study two specific dyes namely MG and Victoria pure blue BO. The data were further processed to study the metabolic fingerprints by means of statistical tools implemented through the Workflow4metabolomics, a collaborative research network for comprehensive metabolomics data pre-processing, statistical analysis, and interpretation.

O-27   A Multi-Class method for the determination and confirmation of Veterinary Antibiotics and Growth Promoters in Aquaculture Products by LC-MS/MS and LC-HRAM-MS

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The Canadian Food Inspection Agency's Dartmouth Laboratory is responsible for regulatory veterinary antibiotic and growth promoter residue analyses for domestic and imported aquaculture products in Canada. Currently the Laboratory employs thirteen single-class, targeted methods to accomplish this mandate. Current trends in trace level analytical methods are focusing on multi-class determination of residues with a single analysis. The Dartmouth Laboratory has developed a method to detect, quantify, and confirm 60 compounds across seven classes of antibiotics (sulfonamides, tetracyclines, macrolides, amphenicols, nitromidazoles. quinolones & triphenylmethane dyes) and two classes of growth promoters (stilbenes & steroids) using Ultra High Performance Liquid Chromatography with Tandem Mass Spectrometry (UHPLC-MS/MS). Single laboratory method validation data from two different triple-quadrupole instruments will be presented with confirmation criteria utilizing "unit" and high resolution (Orbitrap) mass detectors. The advantages and disadvantages of both confirmatory approaches will be discussed as well as the overall efficiency gains for the Laboratory. Specific challenges of this method compared to targeted methods, including those related to detection limits, will be discussed. Future work will be described, including a survey of 100 samples to test the method in a simulated routine monitoring environment.

O-28   Analysis of pesticides, POPs and emerging contaminants in catfish

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US seafood import was record high at 2.8 million metric tons in 2016, with catfish import having the highest increase at 20%. Most of the catfish is imported to the US from Vietnam, China and Cambodia. While catfish is a valuable source of vitamin D[2] and omega-3 fatty acids, there is also a concern about its contamination with lipophilic organic contaminants. We have developed and validated a multi-class, multiresidue method for analysis of 200 organic contaminants,

including persistent organic pollutants (POPs): *i.e.* stable toxic organochlorine pesticides, polycyclic biphenyls (PCBs), and polybrominated diphenyl ethers (PBDEs), as well as current use pesticides, polycyclic aromatic hydrocarbons (PAHs) and emerging flame retardants. Samples of catfish muscle were extracted with acetonitrile using QuEChERS, and an aliquot of the extract was subjected to dispersive solid phase extraction (d-SPE) cleanup, following by analysis with low pressure (LP) vacuum outlet gas chromatography (GC) tandem mass spectrometry (MS/MS). The method was validated at four spiking levels (1, 5, 50 and 100 ng/g), demonstrating satisfactory recoveries (70-120%) and RSDs (<20%) for most of the analytes. The validated method was applied to catfish samples from different countries – USA, China, Vietnam and Cambodia. Residual amounts of PAHs: naphthalene, acenaphthene, fluorene, phenanthrene, anthracene, and fluoranthene were found. Among pesticides, DDT metabolites: DDE and DDD, and chlorpyrifos, heptachlor, dieldrin, nonachlor, and chlordane were found. Two commonly used flame retardants - dechlorane plus and triphenyl phosphate were also found in catfish muscle.

O-29   Targeted and no-targeted approaches to the determination of microcystins and other cyanotoxins in water using combinations of one and two dimensional liquid chromatography (1D & 2D LC) and tandem quadrupole (TQ), quadrupole time of flight (QTof) and ion mobility quadrupole time of flight (IMS QTof) mass spectrometry

Xavier Ortiz[1] Eva Korenkova,[1] Karen MacPherson,[1] Eric Reiner,[1] Julie Degryse,[2] Adam Ladak,[3] Lauren Mullin,[3] Claude Mallet,[3] John Vukovic,[3] Marijn Van Hulle ,[4] Simon Hird;[5]

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Cyanobacteria (blue-green algae), photosynthetic organisms found in both marine and freshwater environments, produce secondary metabolites including microcystins, some of which are toxic to higher organisms after ingestion or contact with water, causing sickness and public concern. Over 100 different microcystins have been reported to date of which microcystin-LR (MC-LR) is the most common. The WHO provisional guideline value for MC-LR (1 µg/L), which was deemed protective of total microcystins, has been adopted as a basis for national standards or guideline values for drinking water in many countries. Some countries also set limits for microcystins in water-bodies used for recreation. To check compliance with these limits, water testing laboratories have turned to using LC-MS/MS on TQ instruments for the determination of a range of cyanotoxins. This method provided fast and reliable determination of targeted cyanotoxins in surface and drinking water samples at concentrations well below the WHO provisional guideline value for MC-LR of 1 µg/L. Direct injection of the water samples negated the need for extraction. To overcome matrix effects, in the absence of internal standards, quantitation was provided by standard addition.
However, the scope of the LC-MS/MS method was limited to the toxins for which reference standards are commercially available. Approaches for non-targeted analysis of microcystins are described, including the use of 2D LC for on-line sample preparation, data dependant analysis (DDA) on a QTof to extend the scope to 100 microcystins in water and the benefits of ion mobility for screening novel mycrocystins in algal blooms using IMS QTof.

O-30   Software Automation Strategies for Improving Quality and Throughput of Highly Complex LC-MS/MS Analysis

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Improvements in physical elements of LC-MS/MS analysis, such as automated sample preparation methodologies or highly performant/robust instrumentation, have proven valuable in meeting the needs of high quality analysis in high throughput conditions. The resulting datasets, however, can be extremely complex, often requiring many hours of manual inspection in order to convert instrument datafiles into meaningful results (the manual intervention of which can increase technical and regulatory burden). Advances in instrument data systems have provided some assistance, but the fundamental issue often lies at levels below the conventional "flagging" metrics, namely, the chromatographic complexity itself (e.g. unresolved peaks; low concentration analytes in abundant chemical matrix).
Sophisticated peak modeling techniques have been employed to address this issue. The use of an Exponentially Modified Gaussian (EMG) model approach is presented, which allows for deconvolution of even complex sets of chromatographically unresolved mixtures. Data are presented which illustrate the differences achievable in real-world samples, and compares nominal EMG peak processing to conventional integration techniques (with and without manual intervention).

The resultant datasets also lend themselves to cross-batch aggregation, for the purposes of the automated generation of long-running quality control charts and other measure of scientific and business performance. A set of such metrics, taken from an actual laboratory, is presented as a way to illustrate the investigative and quality process improvement capabilities which can be garnered when utilizing modeling and robust statistical methodologies. The example shows the identification followed by the quality and throughput improvement of a particular set of quality issues.

O-31   Comparing Effect-Directed Analyses of Endocrine Disrupting Compounds Using Liquid Chromatography-Mass Spectrometry and ER-CALUX

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Recent developments applying effect-based tools (EBT) for environmental impact screening have been significant. There is good evidence that in-vitro single mode activity bioassays such as Estrogen Receptor Chemical-Activated LUciferase gene eXpression (ER-CALUX) can be used to complement advanced chemical analysis. The ER-CALUX bioassay can provide an estimate of the cumulative chemical effect that a group of contaminants may exert on specific environmental receptors. Di Paolo et al. also demonstrated that ER-CALUX can achieve consistent results from an inter-laboratory study involving 11 laboratories.; therefore, allowing for the exchange of results between various monitoring agencies to optimize resources. In this study, duplicate field sludge samples collected from four local waste water treatment plants were prepared and analyzed by ER-CALUX. Samples with ER-CALUX results that exceeded ER-CALUX threshold values were further analyzed using two different liquid chromatography-tandem mass spectrometry (LC-MS/MS) systems for 57 target chemicals including β-estradiol(β-E2) and 17-α-ethinylestradiol(α-EE2). Quantitative LC-MS/MS results were then correlated to the ER-CALUX results. Initial LC-MS/MS results showed method detection limits (MDL) of β-E2 and α-EE2, currently at 500 and 50 ng/L for the two LC-MS/MS systems used, is inferior to MDL of ER-CALUX bioassay (estimated in the low pg/L ranges) and cannot be used to sufficiently interpret results obtained from the ER-CALUX. Other target compounds such as progesterone and estrone were also found by LC-MS/MS, and might be used to correlate the effects observed by the ER-CALUX. Method improvements to reduce MDLs for LC-MS/MS analysis for β-E2 and α-EE2 to achieve better correlation are currently underway.

O-32   Integrated Capillary Electrophoresis and Electrospray Ionization Mass Spectrometry for the Analysis of Polar Pesticide Residues in Tree Nuts and Comparison with Liquid Chromatography

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Methods to quantitate residues of highly polar pesticides have been of great interest recently for a number of reasons including: highly publicized concerns about health effects (i.e. – glyphosate), MRL violations caused by biogenic and anthropogenic interferents (i.e. – fosetyl-aluminum, phosphonic acid and phosphoric acid), and just the large amounts of these compounds that are used each year (i.e. – glyphosate, glufosinate and paraquat. Analysis with LC-MSMS using stationary phases types such as: graphitized carbon, ion exchange, HILIC, mixed, and polar phases as well as IC-MSMS, have been reported, but these methods can have disadvantages such as matrix suppression, poor retention time stability (especially when analyzing residues in nut matrices), and a limited number of analytes (and analyte classes) that can be analyzed within a single method. There is no polar counterpart to the reversed phase multi-residue screens capable of testing for dozens to hundreds of compounds (for example, the QuPPe method for polar compounds has 8 separate analysis methods using at least 5 different LC columns).

Capillary electrophoresis, coupled to tandem mass spectrometry (CESI-MSMS), has been found to be a promising alternative to LC-MSMS analysis. Preliminary results from an inter-laboratory study using CESI- and LC-MSMS for the analysis of glyphosate, AMPA, glufosinate, fosetyl-Al, phosphonic and phosphoric acids, ethephon, paraquat, diquat, mepiquat, and chlormequat in tree nut matrices (< 10 minute separation time) show that show that CESI-MSMS can be highly effective in the separation of charged pesticides with highly stable retention times virtually no matrix suppression.

O-33   Collection and Analysis of Plant Nectar and Pollen and Honey Bee Collected Pollen at Ornamental Nurseries

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Protecting pollinators from hazardous pesticide exposure from products used by the ornamental horticulture industry requires understanding residue dynamics. How does the timing and mode of application of systemic pesticides translate to concentrations of those pesticides in pollen and nectar? To answer this question we have begun to study pesticide residues in pollen and nectar from plants being grown by nurseries in Connecticut. Collection of these materials can be quite laborious, and concentrations can be in the parts per billion range; so it is important to have analytical methods that are sensitive and specific even with small sample sizes. We have collected pollen and nectar following experimental applications of neonicotinoid pesticides to several model plant species and analyzed them with a modified version of QuEChERS followed by LC/MS-MS. The results show that the compounds do translocate into the nectar and pollen and can reach concentrations of toxicological significance.   At the same time we are also examining bee-collected pollen from hives located within ornamental nurseries to look at actual pesticide exposure. Details on sample collection methods, the analytical protocols and preliminary data will be presented.

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POSTER ABSTRACTS

P-3   Examining Bioaccumulation of Select Contaminants of Emerging Concern in Clams, Mussels and Oysters

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By 2050 70% of human populations will reside in urban areas. High population density results in concentrated chemical use, which leads to exposures for human populations and ecosystems receiving waste streams within and from these urban centers. In developing nations, where many megacities will continue to emerge, access to chemical products is occurring faster than environmental management systems are being implemented. By 2050 it is estimated that global food production must increase by 50%. Aquaculture, which is growing 3-5 times faster than land-based agriculture, will play an important role to meet these needs. Unfortunately, 80% of the global sewage production is not treated, but returned to the environment and subject to reuse. These non-traditional waters are being recycled for agriculture, including aquaculture in peri-urban areas. We are examining bioaccumulation of contaminants of emerging concern (CEC) in bivalves, primarily using isotope dilution LC-MS/MS. Additional analyses are performed for persistent organic pollutants and pesticides. Here, we report findings from our efforts in the USA and Hong Kong, where targeted analysis of CECs was performed in clams, mussels and/or oysters obtained from urban inland and coastal aquatic systems. For example, we examined CECs in green-lipped mussels and oysters collected adjacent to point source municipal wastewater and landfill leachate effluent discharges, respectively, in Hong Kong. We observed erythromycin, sertraline, carbamazepine, diphenhydramine and the drug of abuse ketamine above statistically determined MDLs at low μg/kg levels. These observations may support waste stream, water resource and food safety assessment and management in specific regions.

P-4   Combining headspace solid phase microextraction and surface enhanced Raman scattering to detect the pesticide fonofos in apple juice

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Gas chromatography–mass spectrometry (GC-MS) is conventionally used for detecting vaporizable or volatile compounds. However, it is time consuming and requires complex sample preparation and trained lab skills. We developed an innovative approach that couples headspace solid phase microextraction with surface-enhanced Raman spectroscopy (SERS) for a volatile pesticide (i.e. fonofos) detection in a liquid complex matrix (i.e. apple juice). A gold nanoparticles coated fiber was fabricated by reducing gold (III) on a chemically etched stainless-steel wire. The fabricated fibers were then tested in a headspace method and a dip method for solid phase microextraction and followed by SERS detection of fonofos in water and apple juice samples. Using the headspace method, we can detect as low as 5 ppb fonofos in water and apple juice, as compared to the dip method which can only reach 10 ppb in water and 50 ppb in apple juice. This study demonstrated the potential capability of the method in rapid (within 30 min) and sensitive detection volatile and vaporizable compounds in complex matrices. Future work is needed to optimize the fiber by minimizing the signal variation and test in a variety of target compounds and matrices.

P-5   Legacy Contaminant Fate in the Gulf of Mexico

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The Mississippi river and its tributaries pass through 31 states, and ultimately empty into the Gulf of Mexico, with it many legacy chlorinated hydrocarbon contaminants. Legacy contaminants such as PCBs and DDT have been banned for over 30 years but often remain in the environment, as do their degradation products. These contaminants enter the environment through runoff from agricultural lands, the destruction/disposal of industrial plants and equipment, from emissions from construction materials, and old electrical equipment. Sediment samples from five locations were collected during a NSF cruise on 26 August 2016-7 September 2016 using a multicore collection method. Samples were taken along a transect extending from the mouth of the Mississippi River to a deep offshore location where no

contaminants would be expected to be found. Samples were divided into subsamples by being cut into twelve 1 cm slices. Chlorinated hydrocarbons will persist in sediments however little information has been generated about their presence in lower Mississippi River sediments or coastal sediments near the mouth of the Mississippi River. Accelerated Solvent Extraction and Gas Chromatography-Mass Spectrometry will be used to detect and quantify legacy contaminants amounts in sediment core samples collected at the 5 locations within the Gulf of Mexico.

P-6   Organophosphate Mediated Inhibition of Human Acetylcholinesterase Requires Tryptophan (Trp86) On Its Active Site: An In-Silico Analysis

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Organophosphates (OPs) were screened by molecular docking with human acetylcholinesterase (*h*AChE) using Glide docking module of Schrodinger suites. Information of *h*AChE inhibition by OPs is limited as co-crystal structure between two are not available in Protein Data Bank. In initial screening Trp86 was found to be involved in maximum -Cation interaction on anionic subsite of *h*AChE other than Ser203 (Catalytic site). Site directed mutagenesis and molecular dynamic simulation (MDS) approach were used to explore mechanistic insight of *h*ACHE inhibition by OPs. More than 200 OP molecules were investigated using with extra precision glide docking phoxim ethyl phosphonate (PEP) lead among 200 OPs by interacting with Trp86, Gly121 and Ser203. Site directed mutagenesis at Trp86 (Trp86 to Ala86) shown the deterioration of the binding site in terms of size reduction, loss of electrostatic and geometric stabilization in binding cavity and significant reduction in binding of OPs in preferred orientation. Dock score of both wild and mutated *h*AChE shows a perfect qualitative agreement (R^2=64.1%) towards the study. Molecular dynamic simulation (GROMACS 4.5.5) of *h*AChE-PEP complex for 4 X 104 pico-second with SPC16 water system at 310K temperature explained the evident role of Trp-86 in stabilizing the ligand at P-site of the enzyme. Asp74 and Tyr124 were noticed in conveying H-bonds. Trp86 and Ser203 have shown consistent and better stability of bond based on distance factor (between residues and ligand). As residue Trp86plays significant role, it can be further explored for antidotes development in case of human poisoning.

P-7   Quantification of Toxic Metals and Antioxidants in Hot Pepper, *Capsicum* spp.

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Cd, Pb, and Ni accumulation in pepper fruits of *Capsicum* spp. of plants grown under field conditions were monitored using two extraction procedures, nitric acid solution for total metal extraction and $CaCl_2$ solution for extraction of metal ions. Inductively coupled plasma mass spectrometer (ICP-MS) was used to determine the concentration of these three metals in pepper fruits at maturity. Plant Introduction (PI) number 355820 accumulated significant concentrations of Cd (0.47μg g^{-1} dry fruit). PI260522 accumulated the highest concentration of Pb (2.12 μg g^{-1} dry fruit) among the accessions analyzed. In addition, PI238051 contained the greatest level of Ni (17.2 μg g^{-1} dry fruit). On the contrary, the result also revealed that some genotypes could be used as potential sources of ascorbic acid (PI631144, PI439420, PI241676), phenols (PI639661, PI631144), and β-carotene (PI127442). This variability among the genotypes of the same species might enable the identification and/or development of pepper germplasm with high levels of health-promoting properties. We concluded that high toxic metal accumulator genotypes might be useful for remediation of contaminated sites through phytoremediation, whereas genotypes that contain great concentrations of ascorbic acid, phenols and β-carotene might be useful candidates in plant breeding programs seeking food with health promoting properties.

P-8   Analysis of Veterinary Drug Residues in Imported and Domestic Crawfish using Liquid Chromatography Time-Of Flight Mass Spectrometry

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Chloramphenicol and fluoroquinolone antibiotics are included as high enforcement priority drugs by the USFDA. They are not approved for use in aquaculture; however, detectable levels have been found in imported seafood samples. Countries that export crawfish to the U.S., specifically developing countries, are subject to fewer regulations and lower production standards. In order to control disease and parasites associated with poor water quality and low production standards at aquaculture facilities, farmers turn to unapproved drugs, especially since relatively few new veterinary

drugs are approved for aquaculture. Recently there has been limited information on veterinary drug residues in imported crawfish, even though the number of imports is growing each year. In order to find out if crawfish are indeed grown without the use of antibiotics, it is essential to have a method to test samples for multiple veterinary drug residues. The purpose of this investigation was to validate a single liquid chromatography-mass spectrometry (LC/MS) method for ciprofloxacin, enrofloxacin, sarafloxacin, florfenicol, and chloramphenicol in crawfish meat based upon previous FDA methods. An Agilent TOF LC/MS operated in the positive and negative ion mode with polarity switching was used for the analysis of sample extracts. Ciprofloxacin, enrofloxacin, and sarafloxacin were measured in positive ion mode; florfenicol and chloramphenicol in negative ion mode.

P-9   High-Throughput Determination of Neonicotinoid Insecticides in Pollen and Nectar using Liquid Chromatography with Tandem Mass Spectrometry Detection.

Joe Warnick

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The unexplained death of a honey bee population is referred to as Colony Collapse Disorder. Several possible factors including viruses, parasites, poor nutrition, and exposure to neonicotinoid insecticides are being studied. The increase in study related samples has facilitated the need to create a high- throughput workflow for sample analysis. Pollen and nectar are collected from agricultural crops in very small quantities, usually less than 100 mg.   Study requirements for limit of quantitation (LOQ) are also very low (<1 ppb), making extraction of the entire sample necessary. Liquid chromatography with tandem mass spectrometry detection is the technique of choice for analysis of neonicotinoids. The small sample sizes are well suited for extraction and clean up in a 96 well plate format, making processing large numbers of samples possible. This research provides bridging data for a representative neonicotinoid insecticide, thiamethoxam, and its major metabolite clothianidin. Fortification recovery using high-throughput techniques at the LOQ and 10x LOQ levels are in excess of 70% with a relative standard deviation of <20%.   Implementation of these techniques will help laboratories meet the throughput requirements for the increasing number of pollinator health studies to be conducted in the near future.

P-10   Rapid Screening and Determination of Gamma-hydroxybutyric Acid (GHB) in Dietary Supplements and Energy Drinks by GC-MS-MS

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Gamma-hydroxybutyric acid (GHB) has been used illegally for increasing muscle growth and for increasing athletic performance as well as one of the most common "club drugs". An analytical method has been developed for rapidly screening GHB in dietary supplements and energy drinks.   For solid samples (e.g., capsules, tablets, powder), they were extracted by ethyl acetate containing 2% acetic acid, solutions were then centrifuged and filtered with 0.22mm PFTE syringe filters. Aqueous samples (e.g., energy drinks) were directly extracted using ethyl acetate containing 2% acetic acid. For samples with high oil content (e.g., softgels with fish oil, essential oils), they were first extracted by H_2O with 3% acetic acid, then an aliquot of water extracts was extracted again with ethyl acetate containing 2% acetic acid. An aliquot of ethyl acetate was dried at 65°C under a gentle flow of nitrogen. The dry residues of GHB extracts were derivatized using trimethylsilylation and a mixture of BSTFA/TMCS (99:1, v/v) and ethyl acetate at 600W for 1 min in a microwave oven.   The derivatized GHB and deuterated GHB-D6 (as internal standard) were analyzed by an Agilent 7000C QQQ gas chromatography–tandem mass spectrometer in MRM mode with an overall analysis time of 13 min. The limits of detection and limit of quantification for the targeted compounds were below 50 or 100ng/g and average recoveries were greater than 70% for spiked samples. The results showed that the method was suitable for rapidly screening and determining GHB in dietary supplements and energy drinks.

P-11   Detection of Selective Androgenic Receptor Modulators (SARMs) in Dietary Supplements by HPLC-TOF/MS

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The detection of adulterants in supplements has over time become an increasingly important and challenging task. New additions to the World Anti-Doping Agency (WADA) list of prohibited substances, as new classes of compounds are discovered, necessitate new analysis methods. One such class of compounds is the Selective Androgenic Receptor Modulators (SARMs). These novel compounds have androgenic effects on muscle tissue and have been recently used

by athletes intentionally and unintentionally despite none of these compounds gaining regulatory approval for medical use. With these compounds appearing on the grey-market as unapproved supplements and with claims of athletes unknowingly taking adulterated supplements, a robust method for the detection of SARMs in herbal and non-herbal supplements is necessary.

A new method was developed to detect SARMs in botanical matrices by LC-TOF/MS. This analysis is very challenging due to the large variety in chemical structures of these compounds. To address those issues, a High Resolution Mass Spectrometer was used for non-targeted analysis of SARMs in dry botanical ingredients. Spiked with SARMs standards, botanicals were extracted with methanol and the analytes were separated on a C-18 column. The residues were detected in full scan and compared with a custom built database generated using "Agilent All Ions". This approach allowed for a quick confirmation of detected analytes by identifying structural fragments of target molecules.

P-12   Investigation of Antidepressant Load Reduction Following Tertiary Wastewater Disinfection Treatment

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Treated wastewater effluent is a source of emerging contaminants in surface waters, particularly pharmaceutical compounds from human use. In the spring of 2016, two major wastewater reclamation plants (WRPs) in Chicago, IL upgraded treatment to include chlorination/dechlorination at one, and UV-radiation at the other. The objective of this research is to determine the effect these two types of disinfection treatments have on antidepressant pharmaceutical loads in wastewater, and to establish potential byproducts. Antidepressants were studied due to their widespread use, frequent detection in wastewater effluent, and biological activity at low concentrations. Examining the effect of the upgrade on the effluent dominated ecosystem of the Greater Chicago Area will provide useful data for informing environmental engineering best practices in WRPs. Bench studies in deionized water and wastewater were conducted to systematically measure the reactions of hypochlorite with nine anti-depressants and their active metabolites (fluoxetine, norfluoxetine, sertraline, norsertraline, venlafaxine, citalopram, bupropion, duloxetine, and paroxetine) and carbamazepine. Analysis was accomplished using LC-MS/MS along with LC-UV. Both chlorination/dechlorination and UV-radiation conditions were designed to best mimic treatment at the two WRPs in Chicago. Results indicate that antidepressants can undergo reversible, semi-reversible, or irreversible transformations with hypochlorite in both matrices. UV-radiation bench studies were also completed with the selected pharmaceuticals. Degradation by UV-radiation was observed for most of the pharmaceuticals, but transformations for some were on timescales longer than that of typical treatment at the WRP. Disinfection processes appear to be promising at reducing the amount of neuro-endocrine disrupting compounds from wastewater effluent.

P-13   Developing an Air Sampling Program for the Monitoring of Pesticide Drift and Volatilization

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With the increasing close proximity of agricultural lands to schools and communities, concerns with pesticide drift and volatilization have become an ever more pressing issue. The Florida Department of Agriculture and Consumer Services' need for an analytical response to air issues has prompted the Division of Agricultural Environmental Services (AES) to develop air sampling and analysis methodology for both indoor and outdoor environments. AES has participated in two air monitoring studies: the Good Neighbors Practices (GNP) grant, which included air sampling of a cabbage field in Elkton, Florida; and a Bedbug research project in collaboration with the University of Florida. Air pumps, calibrators and other equipment were purchased. Sampling towers for outdoor air collection were constructed by AES from PVC, and analytical methods were developed. Samples for the GNP were collected over a 6-day period. For the Bedbug study, air samples were taken inside an apartment during the heated pesticide treatment and the aeration periods to assist with reentry time modeling.

P-14   Comparison between HILIC and RP HPLC column methods for glyphosate analysis with tandem mass spectrometry

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Two chromatographic methods, a HILIC and a RP HPLC were developed and optimized for determination of glyphosate and its metabolites N-acetyl glyphosate, N-acetyl-aminomethylphosphonic acid and aminomethylphosphonic acid (AMPA). Both methods were sensitive to detect 1 ng/mL level of the compounds in solvent. The dynamic linear range is between 1 ng/mL and 10,000 ng/mL. The precision and accuracy were met or exceed most of the regulatory analysis requirements. However, for the analysis of the extractions from food matrices, the HILIC method could not separate some interference peak. The RP method with ion-paring technique can separate the interference peak. The conclusion was that the ion-paring RP method is suitable for food analysis. A multi-laboratory validation is going on based on the method.

P-15   Quantitative analysis of colistin in egg, milk and animal tissues by LC-MS/MS

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Colistin is a kind of antibiotics used to treat infections in animals. It is also used to treat human infections by its effective antimicrobial ability, and is considered a drug of last resort in human medicine. In 2015, the plasmid-mediated colistin resistance gene, *mcr-1*, was discovered. To reduce the probability of *mcr-1* transfer between different bacteria and between humans and animals, many countries limited the use of colistin. As this result, we developed a simple, quick and selective method for colistin analysis in variety of livestock matrices (chicken, bovine, kidney, liver, egg, and milk) by liquid chromatography coupled to tandem mass spectrometry. The extraction procedure was applied by using 1M or 6M HCl to liberate the analytes from proteins and followed by using solid phase extraction cartridge to clean-up extracts. Polymyxin B was used as internal standard (IS) in this study to correct the loss during operation and matrix effect. The calibration curves showed good linearity over the concentration range of 5-200 ng/mL with determination coefficients ≥ 0.995. The mean recoveries were in the range 74-112% with coefficient of variation < 14% for all samples. The limits of quantitation ranged from 24 to 150 ng/g (mL). This developed method was applied to market samples for monitoring colistin in foods.

P-16   Development and Validation of a Method for Glyphosate and AMPA Analysis in Various Food Commodities Using Derivatization and LC-MS/MS

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A simple, high-throughput method was developed and validated for the quantification and identification of glyphosate [N-(phosphonomethyl)glycine] and its metabolite AMPA (aminomethylphosphonic acid) in a large number of raw and processed agricultural commodities. Stable isotope labeled internal standards and a derivatization procedure with FMOC (fluorenylmethyloxycarbonyl chloride) were employed for the analysis by liquid chromatography - tandem mass spectrometry. The goals of the development were to provide a single analytical method for use on all matrices, with a target limit of quantitation (LOQ) of 10-50ppb for both glyphosate and AMPA. Attempts to achieve the development goals required optimization of the derivatizaton reaction, chromatography development, extraction and cleanup options, and characterization of matrix-specific issues. Various challenges experienced during the development project will be discussed in detail.

P-17   Residue analysis of Glyphosate, Glufosinate and metabolites using anion exchange clean-up, TMOA derivatization and LC/MS/MS

Kathryn Hunter, Michael Conway

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A rapid method for analysis of Glyphosate, Glufosinate and metabolites (AMPA and MPPA) in agricultural commodities using liquid chromatography and triple quadrupole mass spectroscopy (LC-MSMS) has been paired to a scaled-down version of the extraction and clean-up procedure developed by Tseng et.al (J. Agric. Food Chem. 2004, 52, 4057-4063). A weighed portion of sample is extracted with water and an aliquot is cleaned using strong anion exchange (AG-1X8, acetate) resin. Following vacuum concentration, the residue is derivatized with Trimethylorthoacetate (TMOA), concentrated using nitrogen evaporation, and re-dissolved in water/methanol (4:1).
A Waters® HSS-T3 (C18) column was used to chromatograph the N-acetylated and methylated derivatives which were detected using a Waters Quattro Premier XE (ES positive, MRM). Due to the selectivity and sensitivity of LC/MS/MS, the florisil clean-up (described in the original method for analysis using GC-FPD) was found to be unnecessary for most

matrices. Stable isotopes of all four compounds (commercially available from Toronto Research Chemicals) were used to correct for sample extraction and derivatization efficiency as well as account for differences in matrix suppression. Limits of Quantification for each analyte range from 0.01 to 0.05 ppm, depending on the analyte and matrix. The method has been in routine use for over a year and recovery trends for a variety of matrices will be presented.

P-18   Development of LC-MS/MS method for analyzing glyphosate in agricultural streams in Ohio

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Glyphosate is a broad spectrum, non-selective herbicide that is widely used in agriculture such as corn and soybean. After the introduction of genetically modified crops, the use of glyphosate has significantly increased. According to National Agriculture Statistics Service, glyphosate use in agriculture in the state of Ohio has increased 20 times from 15 X 104 kg in 1990 to 30 X 105 kg in 2015. Glyphosate is known to have low toxicity and high maximum residue level (MRL) of 5 μg/g to 20 μg/g in corn and soybean, much higher than other pesticides. Due to its increasing use, and potential human health risks, monitoring of glyphosate in agricultural watershed is warranted. Glyphosate has high polarity, high water solubility, retention problems and co-elution of other interfering ions on reverse-phased column along with absence of chromophore or fluorophore. This makes it difficult to extract glyphosate from environmental matrices using regular solid phase extraction (SPE) systems and causes challenges in measuring glyphosate using direct GC/MS or reversed-phase LC-MS/MS techniques without pre- or post- column derivatization. In the process of our method development using triple quadrupole mass spectrometer, we have tested several SPE cartridges and disks with sorbing materials having a wide range of retention properties and polarity. We have also tested a C18 reverse-phased column, a porous graphite LC column, and a mixed mode column. Results from efficiency and recovery studies of various SPE systems along with the suitability of the LC columns for glyphosate analyses will be presented.

P-19   Determination of Glyphosate Residues in Dry Botanical Matrices by Solid Phase Extraction-Liquid Chromatography-Tandem Mass Spectrometry

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Glyphosate is one of the most common, broad spectrum herbicides in the world. It is considered to be environmentally and toxicologically safe; however, its wide use has brought up concerns regarding ecological and human health.
Due to the increased application of glyphosate to fields, plants are at risk of accumulating glyphosate and its metabolites. Since the presence of glyphosate in dried botanicals has become a concern for manufacturers of dietary supplements, the development of a simple, efficient and sensitive method was needed.
A method has been established utilizing an aqueous extraction of glyphosate and its metabolites (AMPA, MPPA and glufosinate) from botanical matrices, followed by a dual Solid Phase Extraction (SPE) sample clean up to remove the interfering matrix. Full chromatographic separation was achieved on an ion exchange column using a weakly acidic mobile phase. The analytes were identified and quantitated by Agilent 6470 mass spectrometer with the use of isotope-labelled internal standards. Established limits of detection and quantitation for glyphosate in the matrix were 2 ng/g and 5 ng/g, respectively, which are well below regulatory limits in the EU. This method can be applied to various dried botanical matrices where glyphosate contamination remains a concern.

P-20   A Simplified Method for Identification and Quantification of Two Capsaicinoids in Hot Pepper Fruits

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Two capsaicinoids (capsaicin; CAP and dihydrocapsaicin; DHCAP) in hot pepper, *Capsicum* spp. create a desirable spice and a valued international commodity. CAP and DHCAP were extracted from twenty-six genotypes (accessions) of fresh pepper fruits grown under field conditions using methanol, then passed through a syringe filter discs, and injected into a gas chromatograph (GC) equipped with a nitrogen-phosphorus detector (NPD). CAP and DHCAP were the predominant capsaicinoids in the crude fruit extracts, although concentrations of each varied among genotypes tested. No one accession contained the greatest concentration of both. Nordihydrocapsaicin was always present at a very low

concentration when compared to CAP and DHCAP. Analysis of fruit extracts using mass spectrometry indicated the incidence of two molecular fragments at m/z 305 and 307 that correspond to CAP and DHCAP, respectively. The results revealed that plant identification (PI) 631144 from *C. frutescens* contained the greatest concentration of CAP (323 µg g⁻¹ fresh fruit), whereas PI 123474 from *C. annuum* contained the greatest concentrations of DHCAP (205 µg g⁻¹ fresh fruit) among the accessions tested.

P-21   Validation of analytical methods for the Determination of test substances in variety of matrices

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Residue analytical methods are required to support various studies required for pesticide registration including: magnitude of residue, environmental fate, ecotoxicology, toxicology, and operator or worker exposure studies. Current residue analytical guidelines include SANCO/3029/99 rev.4 in support of pre-registration of pesticides, SANCO/825/00 rev.8.1 for post-registration monitoring and control of pesticides, and OPPTS 860.1340 for residue analytical methods. The objective of validated methods are their use for quantitative measurements of the active ingredient (AI) and its metabolites, or the degradation products produced from the AI in a wide variety of matrices, including plant materials, soil, water, animal feed and animal tissues. In order to have global applicability for pesticide registration, especially in Japan, Australia, and European countries, method validation following the above SANCO guidelines are required. This poster will discuss the detailed requirements for conducting a method validation based on the various SANCO and EPA guidelines with focus on the fortification levels, number of replications, primary/confirmatory methods, and matrix investigations. Common analytical instrumentation used for method validations include GC-ECD/MSD, HPLC-UV/DAD, and LC-MS/MS. Implications with respect to recovery repeatability, selectivity and confirmation using these various instrumentations will be presented. In some cases, confirmation by an independent analytical technique may also be needed. Independent laboratory validation (ILV) will be discussed since an ILV is generally triggered by new method validations.

P-22   Development of an Immunochromatographic Assay for the Detection of Feed Additive Zilpaterol

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Zilpaterol is a β-adrenergic agonist feed additive approved in the United States to increase weight gain and improve feed efficiency of cattle. Countries which ban β-adrenergic agonist feed additives can reject imported beef products that contain zilpaterol. Therefore, efficient, portable, and user friendly screening assays towards zilpaterol are needed. In this report, an immunochromatographic assay was developed for the rapid detection of zilpaterol. Zilpaterol-butyrate BSA was plotted onto nitrocellulose membrane while colloidal gold labeled with zilpaterol antibody was adsorbed to a glass fiber sample pad. The assay was applied to cattle feed and cattle urine, horse urine, sheep muscle and sheep urine with sensitivities of 23.2, 1.7, 8.8, 2.2, and 1.7 ng/g or ng/mL, respectively. No sample preparation was needed for cattle and sheep urine, but horse urine and cattle feed required dilution; muscle required solvent extraction. The zilpaterol immunochromatographic assay was tested with incurred horse urine, sheep urine, and sheep muscle. Of the 32 incurred sheep urine samples, all zilpaterol treatment samples were correctly identified but 2 false-positives occurred compared to LC-MS results. Incurred horse urine (n=48) containing zilpaterol residue > 10 ppb (~ withdrawal day 6) were correctly identified as positives by the immunochromatographic assay. The immunochromatographic assay correctly identified 0-day withdrawal muscle samples as positive and the control sheep as negative. In conclusion, the developed zilpaterol immunochromatographic assay is useful for on-site, portable testing of urine and feed samples and screening of meat samples.

P-23   Analysis of 10 β-Agonists in pork meat using automated dispersive pipette extraction and LC-MS/MS

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β-Adrenergic agonists are synthetic phenylethanolamine compounds used as bronchodilators and tocolytic agents. While these compounds have medicinal uses in the veterinary field, they are often misused as growth promoters in cattle, swine and other farm animals. In many countries, including European Union countries and China, these drugs have been banned due to their adverse effects on humans, such as food poisoning and cardiovascular and central nervous diseases. Regulations have either not set a maximum residue limit or have a zero-tolerance level for these compounds. An analytical procedure for the analysis of 10 β-adrenergic agonists (cimaterol, terbutaline, salbutamol, isoxsuprine, ractopamine, cimbuterol, clenbuterol, brombuterol, mabuterol and mapenterol) in pork meat was developed and validated using LC-MS/MS. An automated dispersive pipette extraction (DPX) was employed on a Hamilton Microlab® NIMBUS96® platform to extract the analytes of interest prior to LC-MS/MS analysis. The extraction time was less than 20 min with a total LC-MS/MS run time of 9.6 min. The method was fully validated in accordance with the international guidelines (European Commission Decision 2002/657/EC and National Standards of People's Republic of China, GB/T 22286-2008) for limit of detection, limit of quantitation, carryover, extraction efficiency (85-100%), matrix effects (2-25%), linearity (0.2-50 ng/g), and within and between-run precision (CV <21%). The proposed method can be successfully used in the routine determination of 10 β-adrenergic agonists in pork and as a potential solution for compliance monitoring in regulatory laboratories.

P-24   Analysis of Δ-9-tetrahydrocannabinol in plant material using automated dispersive pipette extraction and LC-MS/MS

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Marijuana is the most widely abused drug in the United States. Delta-9-tetrahydrocannabinol (THC) is the main psychoactive ingredient in cannabis. In states where marijuana is legal, analysis of THC in plant material is helpful for quality control prior to sale. In states where marijuana is illegal, analysis of THC in plant material is required for all suspected seized plant material. Therefore, the need for fast robust methods of evaluating THC content continues to rise with the increase in cannabis use. The chemical complexity of marijuana plant material and subsequent extracts necessitate solid phase extraction prior to LC-MS/MS analysis. Current methods either have very laborious sample preparation methods or lack them altogether. An analytical procedure for the determination of THC in plant material was developed using dispersive pipette extraction (DPX) on a Hamilton Microlab® NIMBUS96® platform followed by LC-MS/MS analysis. The DPX extraction focuses on minimal sorbent for reduced matrix effects and reasonable levels of THC to avoid saturating the detector. The automated extraction method can process up to 96 samples simultaneously in under 15 minutes thereby minimizing between sample variability and maximizing throughput. The versatility of the sample preparation method could allow for its use with a variety of matrices including many edible forms of cannabis for both qualitative and quantitative analysis. The proposed method is an ideal automated workflow solution for determining THC content in plant material.

P-25   The Analysis of Water for Perfluorinated Compounds using Automated Solid Phase Extraction

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Perfluorinated compounds (PFCs) are of increasing concern as they are detected in environmental and human samples. Originally thought to be inert compounds, they are long-lived and may cause tumors and endocrine effects. They bioaccumulate so continued exposure may be especially hazardous.   The measurement of PFCs was included in the Unregulated Contaminant Monitoring Program 3 (UCMR-3) and occurrence evaluated in drinking water across the US. US EPA Method 537, which passes 250 mL of water through a cartridge and subsequent analysis with LC/MS/MS, was developed to support this effort. As reports of PFC contamination continue to draw headlines, the need for a simple and automated analysis becomes more critical.
This work evaluates the development of automated methodology for EPA 537. Background levels and the need to develop a system that will minimize contamination will be discussed. A range of water samples will be analyzed and challenges and results presented.

P-26   A Novel Solution to the Analysis of Highly Complex Environmental Samples

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Gas chromatography coupled with mass spectrometry provides some of the best analytical tools which combine selectivity, sensitivity, reliability, and information capacity for both targeted and non-targeted methods of environmental analysis. The GC-MS identification of known compounds of interest and the structural elucidation of unknown compounds becomes considerably more reliable if accompanied by accurate mass measurements while using High Resolution Mass Spectrometry (HRMS). When analyzing real-life samples in a complex matrix, a high number of analytes of interest with a wide range of concentrations are likely present. Consequently, a significant increase in chromatographic peak capacity is required which can be realized by the use of comprehensive GCxGC. An ultra-high resolution time-of-flight mass spectrometer with enhanced sensitivity from LECO was used to analyze various residue and environmental samples. Select examples of pesticide residue matrix spiked samples along with some cloud and rain water samples were analyzed to show a variety of complex environmental sample types. Examples will be shown of how the use of this instrument provides highly reliable data suitable for automatic accurate spectral deconvolution of coeluting analytes present in samples with a wide concentration range. The addition of the GCxGC technique increases the separation power by allowing chromatographic separation of closely eluting constituents, thus making analyte identification more reliable and the comprehensive analysis of environmental samples more realistic.

P-27   Identification and Quantitation of Pesticides in Hops Extracts with a Benchtop GC-TOFMS

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Due to their matrix complexity hops have traditionally been a difficult commodity to monitor for pesticide use. The enormous growth in the craft brewing sector along with the popularity of IPA style beers has led to an increased demand for hops. It is anticipated that the demand for routine pesticide monitoring will likewise continue to rise. In this study gas chromatography was combined with time-of-flight mass spectrometry (TOFMS) to detect commonly monitored pesticides as well as screen for new and emerging pesticides. Whole leaf hops and extracted hop resin were extracted using a combination of QuECHERs and solid phase extraction (SPE) cartridges.

The extracts were injected onto an Agilent 7890B gas chromatograph (GC) fitted with an Agilent Multimode Inlet (MMI) and a standard 30m x 0.25mm ID x 0.25µm Rxi-5SilMS column coupled to a LECO Pegasus BT-TOFMS. The data was collected at 20 spectra per second with a mass range of 35-520 m/z. Identification of incurred pesticides was established by matching deconvoluted spectra to standard MS libraries coupled with software integrated retention index (RI) screening.

Calibration curves were established for incurred pesticides and matrix matched standards were prepared for other commonly used hops pesticides. Statistically derived limits of detection (LOD) were calculated for pesticides in matrix and found to be below required reporting limits demonstrating the Pegasus BT-TOFMS to be an effective platform for screening and quantitation of pesticide residues in hops.

P-28   A Flow Based Triple Quadrupole Mass Spectrometer and Its Application in Infant Formula Label Claim Analysis

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We introduce a new triple quadrupole mass spectrometer (Perkinelmer Qsight 220) that utilizes flow based technology to transport ions in the front end. Together with a coaxial flow ion source, self-cleaning interface, unique mass filter, efficient collision cell, and unifield detector, the Perkinelmer Qsight 220 triple quadrupole mass spectrometer enables sensitive and reliable detection of small molecules from different complex matrices.

Infant formula is designed to simulate and substitute human milk to feed babies or infants that are under 12 month of age. Major components of infant formula include proteins, fats, carbohydrates, vitamins, and minerals. Those components need to be accurately measured to make sure intended use can be achieved, therefore ccuracy of the label claim becomes a vital contributor to the brand and value. Here we present a series of applications developed using Qsight 220 LC-MSMS system, including the determination of bovine alpha-lactalbumin, water soluble vitamins (vitamin B), and folic acid in baby formula. The methods were validated and used to test baby formula products collected from market. Most of the label claims were within acceptable target range, with few outliers.

P-29 **Separation of 12 Natural Cannabinoids in Both Methanol and Acetonitrile Mobile Phases**

Scott Krepich, Allen Misa and Jeff Layne

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Recently there has been an increased focus on the analysis of natural cannabinoids. Several classes of cannabinoids exist, each with different physical and psychoactive effects. The amount of each cannabinoid present varies with each strain of cannabis, thus making accurate quantitation very important. Additionally, as the cannabinoids are subsequently added to different matrices, the resulting analytical methods must offer appropriate solvent recommendations to ensure that unwanted precipitation doesn't occur. By utilizing a high performance Kinetex® 2.6 µm Polar C18 column, we've achieved two fast and effective cannabinoid separations of the 12 currently commercially available certified reference materials using either Acetonitrile or Methanol as the organic strong solvent, alongside a high resolution methanol method at neutral pH and a low pressure acetonitrile method. This allows analytical scientists to choose the appropriate solvent system based on their sample set, while also allowing them to utilize either UHPLC or HPLC instrumentation.

P-30 **Complimentary Column Chemistries for Determination Pesticides in Cannabis by LC-MS/MS**

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The expansion of legalized marijuana, both for medical as well as recreational use, has led to an increase in analytical testing needs such as potency, pesticides, terpenes, and residual solvents. And while the murkiness of the regulatory landscape may be at the heart of many of the challenges, market driven solutions are on the rise, particularly in labs with parallel areas of expertise.

Challenges in pesticide testing often arise from a wide range of physical and chemical analyte properties in multiresidue screening along with diverse sample matrix interferences. Sample clean-up with QuEChERS has become widely adopted in food matrices to remove matrix interferences. Alternatively, a dilute and shoot approach might be taken when sensitivity isn't compromised, such as with high sensitive mass spec detection. Both approaches have been implemented successfully for pesticides in cannabis sample testing, which we demonstrate on multiple and complimentary LC column chemistries in HPLC and UHPLC platforms for the 64 pesticides indicated from the Oregon Health Authority. Luna Omega Polar C18 and Kinetex Biphenyl yield excellent retention and peak shape for the challenging and earliest eluting daminozide, while maintaining a solid chromatographic profile throughout the range. These diverse selectivity options on highly efficient and versatile platforms can be an excellent tool to navigate the myriad of established matrix interferences and those to arise.

P-31 **Analysis of Sensory-Active Volatile Phenols in Smoke-Exposed Grapes by Gas Chromatography**

Matt Noestheden[1,2], Katelyn Thiessen[1], Wesley Zandberg[1], Scott Krepich[3] and Allen Misa[3]

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Volatile phenolic (VP) compounds are expressed endogenously in wine grapes and have been shown to increase following on-vine smoke exposure. Depending on their relative concentrations (among other factors) VPs can possess negative organoleptic properties, with sensory descriptors such as 'ashtray' and 'Band-aid' often associated with wines made using smoke-exposed grapes. The evaluation of smoke exposure is a significant quality control issue for vineyards, but also for wineries, as the concentration of VPs may increase during fermentation or aging. With climate models predicting an increase in the frequency of forest/brush fires and the localization of many key wine producing regions near or in areas prone to such fires (e.g., California, British Columbia, Australia, South Africa), it is critical to expand our current understanding of this economically important phenomenon.

The polyethylene glycol Zebron ZB-WAX GC phase resolved all critical pairs of volatile phenolics, including the very challenging cresol positional isomers. Chromatographic resolution is requisite for accurate quantitation of all three cresol isobars, as their structural similarity results in identical product ions when analysed by GC-MS/MS. Similar chromatographic resolution of the cresols was not feasible in a reasonable time scale using a low polarity, dimethylpolysiloxane phase. The obtained resolution of p-cresol and m-cresol will facilitate the accurate characterization of volatile phenolics in smoke-exposed grapes. In turn, this will aid the development of more accurate models for predicting wine quality issues when using smoke-exposed grapes and may also inform remedial and preventative strategies.

P-32   **Separation of 12 Natural Cannabinoids in Both Methanol and Acetonitrile Mobile Phases**

Scott Krepich, Allen Misa and Jeff Layne

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Recently there has been an increased focus on the analysis of natural cannabinoids. Several classes of cannabinoids exist, each with different physical and psychoactive effects. The amount of each cannabinoid present varies with each strain of cannabis, thus making accurate quantitation very important. Additionally, as the cannabinoids are subsequently added to different matrices, the resulting analytical methods must offer appropriate solvent recommendations to ensure that unwanted precipitation doesn't occur. By utilizing a high performance Kinetex® 2.6 μm Polar C18 column, we've achieved two fast and effective cannabinoid separations of the 12 currently commercially available certified reference materials using either Acetonitrile or Methanol as the organic strong solvent, alongside a high resolution methanol method at neutral pH and a low pressure acetonitrile method. This allows analytical scientists to choose the appropriate solvent system based on their sample set, while also allowing them to utilize either UHPLC or HPLC instrumentation.

P-33   **Evaluation of the AutoMate-Q40 for Organochlorine Pesticides in Water**

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Organochlorine Pesticides (OCPs) are a group of persistent organic pollutants (POPs). In which most of these have been prohibited from use. OCPs are grouped into three group's dichlorodipheny ethane (DDT, DDD, and DDE), cyclodiene (aldrin, dieldrin, heptachlor and endosulfan) and chlorocyclohexane (α, β, γ and δ-HCH).
The QuEChERS extraction method is applicable for OCPs in fruits and vegetables. In this application, Teledyne Tekmar wanted to apply a modified QuEChERS extraction for determination of OCPs in water instead of doing a traditional Liquid-Liquid Extraction (LLE).

The goal of this project is to evaluate the performance and versatility of the AutoMate-Q40 for the extraction of OCPs in water by a modified QuEChERS extraction. Gas Chromatograph coupled to a Mass Spectrometer (GC-MS) was employed for the detection of OCPs in water. Quantification was based on matrix-matched calibration curves using linear regression with 1/x weighting.

P-34   **Determination of pesticides and persistent organic pollutants in honey by accelerated solvent extraction and 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, honeybees are subject to a number of viral, bacterial, fungal, and parasitic diseases and infestations. Insecticides, fungicides, and acaricides are used to protect colonies against infestations from hive beetle and parasites. Many pollutants in the environment can also contaminate the bees themselves in addition to their pollen, honey, and other bee products. Pollutants such as organochlorine pesticides (OCs), polychlorobiphenyls (PCBs), organophosphates (OPs), and polybromodiphenylethers (PBDEs) are a particular threat due to their environmental persistence and ability to bioaccumulate in the food chain. Due to the potential toxicity, a comprehensive workflow method for the extraction and analysis of these environmental pollutants is of growing importance to ensure the health and safety of bees and their honey.
Among the available extraction techniques, accelerated solvent extraction (ASE) offers shorter extraction times and reduced solvent consumption. ASE uses high temperatures combined with high pressure. A high temperature allows a higher rate of extraction due to a reduction in viscosity and surface tension, and increases the solubility and diffusion rate into the sample. The method reported here is applicable for the extraction and analysis of four different classes of compounds (6 PCBs, 7 PBDEs, 16 OCs, and 19 OPs) in honey using ASE and GC-MS/MS.

P-35   Multi-residue Pesticide Screening in Cereals using GC-Orbitrap Mass Spectrometry

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Pesticides are used to improve cereal crop yields and to minimize deterioration and pest damage during storage.. However, the widespread use of pesticides and the potential for residues to remain on the final product is of concern to consumers and Governments whose responsibility it is to ensure a safe food supply. For the complete coverage of the hundreds of pesticides in use, routine residue testing requires both liquid and gas chromatographic techniques coupled mass spectrometers (MS). Triple quadrupole mass spectrometers can provide the required sensitivity and selectivity to ensure that maximum residue levels are not exceeded for a list of targeted pesticides. However, targeted MS methods are limited to only detecting pesticides that are measured at the time of acquisition and require careful method optimization and management to ensure selected ion monitoring windows remain viable. The alternative technique of high resolution Orbitrap mass spectrometry provides distinct advantages over low resolution MS/MS techniques and substantially increases the scope of the analysis. With high resolution mass spectrometry (HRMS), the default acquisition mode is non-n targeted (full scan acquisition) making it easier to set-up and manage methods for an unlimited number of pesticides to be monitored in a single injection.

In this study, the performance of the Thermo Scientific™ Exactive™ GC Orbitrap™ GC-MS system was evaluated for the routine analysis of GC-amenable pesticides in cereals (wheat, barley, oat, rye and rice) following SANTE/11945/2015 guidelines. All 105 pesticides were detected at concentrations lower than 10 µg/Kg based on retention time and the main quantifier ion. A total of 90% of the 525 pesticide/matrix combinations were compliant with SANTE guidelines at ≤10 µg/Kg and 96% at ≤20 µg/Kg. Having multiple identification ions and limits of detection below the MRLs increases the confidence in positive detections. The Exactive GC quantitative linearity was assessed using matrix matched standards in rye across a concentration of 10-300 µg/Kg. In all cases, the coefficient of determination (R2) was >0.99 for each pesticide from its LOD value to 300 µg/Kg. A final assessment was made of the peak area repeatability at low analyte concentrations by replicate (n=20) injections at 10 µg/Kg in wheat. All detected pesticides had RSD% of less than 13%, significantly lower than the 20% guideline limit.

P-36   Fast routine analysis of polar ionic pesticides in food samples by suppressed ion chromatography and mass spectrometry

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In the last two decades there has been a huge increase of pesticide residues that can be analysed all together in one or more multi methods using both GC-MS and LC-MS techniques. However, some compounds due to their very special chemical properties such as polar ionic pesticides still have to be measured separately. Traditional methods include derivatization of the target pesticides to enable better extraction and separation or to utilize special chromatographic columns such as Hypercarb (with graphitic carbon phase) or ion pairing reagents. The drawbacks of these conventional LC-MS/MS approaches can be avoided by employing Ion Chromatography (IC), as it has the ability to effectively retain and separate very polar compounds. The use of a triple quadrupole mass spectrometer provides the necessary sensitivity and selectivity required for food matrices.

The presented method demonstrates the analysis of ten polar pesticides, including emerging compounds like Glyphosate, Glufosinate and AMPA in three different food matrices (lettuce, oranges and wheat flour). The sample preparation is very easy and was adopted from the Quick Polar Pesticides (QuPPe) method developed by the European Reference Laboratory responsible for single residue methods EURL-SRM (1). The method was validated in-house according to the European SANTE guidelines 11945/2015. Analytical parameters such as linearity, specificity, LOD, LOQ, precision and accuracy were evaluated using fortified blank food material at three different levels. All tested parameters showed satisfactory results with LOQs below the required legislative limits. The recoveries were in the range from 70 – 120 % for all compounds.

P-37   A Method for the Quantification of Un-derivatized Amino Acids in Sugar Snaps by LC-MS/MS

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Proteins are considered as the "building blocks" of all living organisms. Although, being huge molecules all proteins are composed of only 20 naturally occurring L-amino acids. Out of these twenty, eleven are essential and must be obtained through dietary supplements. In a wide range of food, amino acids are ubiquitously present as free amino acids and can enhance flavor or taste or increase nutritional value. Due to their pivotal role for animals and humans there is a need for techniques that ensure an efficient and reliable determination of amino acids in food (supplements) and feeds.
We have developed an LC-MS/MS method for the analysis of free amino acids present in the food. The analysis was performed on a Thermo Scientific™ Ultimate 3000™ RS LC system coupled to a Thermo Scientific™ TSQ Endura™ mass spectrometer. The chromatographic separation was carried out on a Discovery HS F5-3 column (Phenomenex, USA). Processing of the data was done using the Thermo Scientific TraceFinder 4.1 software.
The LOQs in neat solvent were in the low µg/L range and all correlation coefficients were above 0.995. Sugar snaps were used as example food matrix. Through standard addition it was possible to quantify nineteen out of twenty amino acids from an amount corresponding to 2 µg or less of matrix. The repeatability was very good with %RSD values lying below 15 for 18 out of 20 target analytes. The method presented here offers a quick and simple way for the analysis of 20 L-amino acids without need for any derivatization.

P-38   Routine Quantitative Method of Analysis for Pesticides Using GC-Orbitrap Mass Spectrometry in Accordance with SANTE/11945/2015 Guidance

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The analysis of pesticide residues in food is challenging because of the high number (typically >800) of substances that need to be analyzed in a diverse range of complex matrices, at low cost and with a fast reporting time. In order to achieve this within a routine environment, sensitive and selective LC and GC triple quadrupole MS systems are used. However, when such large numbers of compounds need to be analyzed it is an advantage to employ a system with full scan acquisition providing the performance is similar to that of triple quadrupole techniques. A generic acquisition based on full scan MS is more straightforward and provides additional information compared with multiple reaction monitoring by triple quadrupole mass spectrometry. It also increases the scope of the analysis, as target compounds are selected post acquisition. In order to obtain sufficient selectivity in the full scan mode, high resolution/high mass accuracy MS instruments are required.
In this study, the quantitative performance of the Thermo Scientific™ Exactive™ GC Orbitrap™ GC-MS system was evaluated for the routine analysis of GC-amenable pesticides in leek, orange and tomato matrices. In total 99.3% of the 153 pesticide/matrix combinations were detected below the respective maximum residue limits (MRLs) with excellent linearity and in compliance with the SANTE/11945/2015 method performance criteria. Quantitative linearity was assessed using matrix matched standards across a concentration of 0.5-500 µg/Kg. In all cases, the coefficient of determination (R2) was >0.99 for each pesticide from its LOD to 500 µg/Kg in the three matrices. Importantly, the scope of the analysis is increased by acquisition in full scan with targeted data processing with a compound database. Acquisition using 60,000 FWHM resolving power (at m/z 200) reduces matrix interferences and increases confidence in results when screening for pesticides in complex sample matrices. Sub ppm mass accuracy was achieved for all compounds over a wide concentration range ensuring that compounds are detected with confidence at low and high concentration levels. Repeated injections of a tomato matrix at 10 µg/Kg showed that the system is able to maintain a consistent level of performance (RSD% <6%) over an extended period of time as is demanded by a routine testing laboratories.

P-39   Robust LC-MS Analysis of Pesticides with 1.0 mm ID columns using state of the art UHPLC instrumentation

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UHPLC-MS applications are frequently developed on 2.1 mm I.D. columns at fairly high flow rates in the range of 0.3-1.0 mL/min. Lower flow rates promise better sensitivity and lower detection limits in mass spectrometry due to easier solvent removal and better ionization efficiency. Simply reducing the flow rate on the same column geometry would worsen the separation efficiency and reduce both chromatographic resolution and sensitivity. The use of 1.0 mm I.D. columns instead of 2.1 mm I.D. columns offers the advantage that the same LC performance can be generated at substantially lower flow rates, additionally reducing mobile phase consumption and hazardous waste production. On top, micro-flow UHPLC applications based on 1.0 mm I.D. columns will show a higher sensitivity compared to normal flow UHPLC. When scaling down a method this theoretical increase of sensitivity follows the ratio of the squares of the internal column diameters. However, if UHPLC methods with 1.0 mm I.D. columns are coupled to MS detection, the sensitivity increase can deviate substantially from the theoretical value due to the specific physical chemical properties of the analytes influencing ionization efficiency. Next to the MS, also the fluidic design and the instrument performance of the UHPLC system need to keep pace with the requirements claimed by the use of 1.0 mm ID columns.

In this study, we evaluate the compatibility of the latest state of the art in UHPLC instrumentation and Triple Quadrupole Mass Spectrometry with 1.0 mm I.D. columns for the trace-level analysis of pesticides at (sub-)ppb level.

P-40 Enhancing analytical confidence and detection limits for IC and LC applications by coupling them to single quadrupole mass spectrometry

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Traditional IC and HPLC environmental applications have relied on bulk physical property monitoring detectors: e.g. conductivity (for IC) and UV-Vis or PDA (for HPLC). With ever more increasing regulatory pressure and general tendencies in analytical chemistry to aim at better detection limits and have a higher degree of confidence in analyte identification and quantitation (i.e. reduce false positives and their contribution to quantitation), there is an emerging trend to couple IC and HPLC with mass-selective detectors; antagonized primarily and only by the latter's high cost and perceived difficulty of adoption and operation. We present a newly developed single quadrupole mass spectrometer (MS) for routine applications, including environmental, that incorporates technological innovations in the hardware and software realms to help resolve the two stated problems. Application examples with analytical figures of merit in environmental analysis, analysis of ionic/organic pesticides, and pharmaceuticals are included to demonstrate the synergistic benefit of IC-MS and LC-MS coupling in routine applications. Special attention will be made to the MS hardware design features that allow coupling it with both ion chromatography as well as liquid chromatography, and to the software innovations supporting ease-of-use and ease-of-adoption.

P-41 Determination of Persistent Organic Pollutants in Fish Tissues by Accelerated Solvent Extraction and GC-MS/MS

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Polychlorinated biphenyls (PCBs), organochlorine pesticides (OCPs) and polybrominated diphenyl ethers (PBDEs) belong to a broad family of synthetic organic compounds known as halogenated hydrocarbons. The capacity of the halogenated hydrocarbons to bioaccumulate in fatty tissues and biomagnify up the food chain, in combination with their resistance to degradation and their toxicity, make this class of chemicals a serious threat to environmental and human health. Due to this potential toxicity, the extraction and analysis of halogenated hydrocarbons from matrices such as fish tissue is required by the U.S. EPA. Techniques such as Soxhlet and sonication are used for the extraction of halogenated hydrocarbons from environmental samples prior to their analytical determination. These techniques are, however, very labor intensive and suffer from high solvent consumption. Accelerated solvent extraction was developed to meet the new requirements of increased throughput and reduced solvent usage in sample preparation.

The work presented in the poster demonstrates workflow methods for halogenated hydrocarbon extraction and analysis using GC-MS/MS from fish tissue. An analytical method was developed and applied to evaluate POP residues in tuna samples from different Food and Agricultural Organization areas. The method reported here is applicable for the determination of 29 halogenated hydrocarbons (6 PCBs, 16 OCPs, and 7 PBDEs). The method proved to be simple and rapid, requiring small sample sizes and minimizing solvent consumption, due to use of accelerated solvent extraction with an in-line clean up step.

P-42   Benefits of a novel automated SPME technology for the detection of environmental pollutants at trace level in water

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Solid phase micro-extraction is widely used as a valid solvent-free pre-concentration technique in the analysis of a wide range of pollutants in environmental water samples. However, since its introduction, the technique remained almost unchanged, retaining its limitations in terms of fiber phase volume and mechanical robustness. Mechanical robustness is a key critical challenge to widely adopt automatically this technology. This paper describes an advancement made integrating the fiber within the syringe needle that allows combining the advantages of the standard SPME together with a larger fiber phase volume, and an increased sample throughput and robustness. In this study, an evaluation of this new SPME technology is presented for the determination of the 16 EPA regulated Polycyclic Aromatic Hydrocarbons in water. Parameters optimized for Direct Immersion SPME along with results on sensitivity, repeatability and linearity obtained with a GC-Single Quad MS system are shown. Data demonstrates the enhanced capability of this system to reach detection limits down to pg L-1 level and the improved mechanical reliability, suggesting this new technology as a valid alternative method for the automated and quantitative analysis of PAHs in water as well as for a wide variety of other potential applications.

P-43   Fast routine analysis of polar ionic pesticides in water by suppressed ion chromatography and mass spectrometry

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Presence of polar pesticides and their metabolites in water and foods has become very hot topic in the past couple of years. The most famous representative of this group is a broad-spectrum systematic herbicide glyphosate and its metabolite AMPA. Because of the chemical properties especially high polarity, it is not possible to analyze these compounds with the conventional C18 column. Typically the laboratories use the methods that include derivatization step or special chromatographic columns like the porous graphitic carbon (PGC) based Hypercarb. With both approaches varying method robustness and unreliable results are often reported by routine laboratories. We do present an Ion Chromatography Mass Spectrometry (IC-MS) method for direct analysis of five polar ionic pesticides (Fosetyl-Al, Glufosinate, AMPA, Clopyralid and Glyphosate) in water samples. IC is the preferred separation technique for polar ionic analytes such as anions, cations or ionic metabolites and thanks to the recent development in the hyphenation of IC and MS it enables its unproblematic usage. The method is very easy and fast with no need for a sample preparation. Only for very dirty surface water samples the filtration through the membrane filters is recommended.
The method was validated in-house for three water matrices covering surface, drinking and bottled water. Analytical parameters as linearity, specificity, LOD, LOQ, precision and accuracy were evaluated using fortified blank water samples at three different levels. All tested parameters showed satisfactory results with LOQs well below required legislative limits.

P-44   Time saving sample prep for the analysis of pesticide residues in Cannabis by LC-MS/MS

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Pesticide analysis of cannabis leaves and finished goods is becoming increasingly important as many states are legalizing it for medicinal and recreational purposes. Dosing methods include smoking/vaporizing and edibles but cannabis is still a Schedule 1 illegal drug and therefore have no FDA testing guidelines. Trace levels of pesticides can be incurred during cultivation or inhaled from dried pesticides on the cannabis. This study evaluates the sample preparation aspect for LC-MS/MS analysis of a panel of pesticides. We compared SPE, QuEChERS, and eXtreme|FV for recovery, sample prep time, and cost.

P-45   Pesticide Transformation Products: Markers of Unauthorized Application in Organic Wine Production

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Modern pesticides can degrade after their application to variety of transformation products (TPs). For this reason the parent compounds might not be detected in the tested matrices / final products. Contrary to conventional agriculture, in organic farming the usage of synthetic pesticides is restricted by the directive 834/2007/ES and 889/2008/ES where only a few, mainly natural pesticides, are permitted. In this case the analysis of pesticide residues belongs to the important tools used in the authenticity control of organic farming products. The analysis of TPs seems to be a promising alternative enabling to prove the application of (unauthorized) pesticides or even to distinguish between its unauthorized use from any unintentional contamination of crops.
The presented study was focused on the determination of pesticide residues and their TPs in vine products (Vitis vinifera - leaves, grapes, musts and wine). An extraction procedure was based on the original version of the QuEChERS method. Crude extracts were analysed by ultra-high-performance liquid chromatography coupled to high resolution mass spectrometry (Quadrupole-Time of Flight spectrometer; Agilent Ion-Mobility Q-TOF 6560, USA) in positive and negative mode. We aimed to describe the dynamics of degradation of parent compounds and formation of TPs during vine growing and maturation and wine making process. Several transformation products originating from dimethomorph, fenhexamid, iprovalicarb, metrafenon, pyraclostrobin, spiroxamine and tebuconazole were identified in vine leaves, grapes, musts and wine. The main identified pesticide transformation routes were oxidation, hydrolysis and conjugation.

P-46   Rapid Enzyme Hydrolysis Using a novel recombinant b-glucuronidase in β-agonists Meat Analysis

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β-agonist compounds are widely used bronchodilator for asthma treatment in human; they are also used as growth promoters in food-producing animals. Misuse of numerous β-agonist drugs for their growth promoting effects in livestock production requires significant regulatory enforcement activities worldwide. These compounds are highly metabolized therefore efficient hydrolysis process is critical to liberate the parent β-agonists from their glucuronide conjugates. β-glucuronidase enzymes can have varying degrees of hydrolysis efficiency based on which matrices are being tested. Two β-glucuronidase enzymes were compared and optimized to achieve the most efficient hydrolysis of beta-agonists in meat matrices. Enzyme concentration, incubation time, and incubation temperature were varied in the hydrolysis studies of enzymes from Helix pomatia (Sigma) and recombinant IMCSzyme® (IMCS). The effects of these variances will be presented. The comparison between two different enzyme sources indicated IMCSzyme had comparable or better recoveries of the glucuronide metabolites in meat samples despite the decrease in enzyme volume and incubation time. The study demonstrated that IMCSzyme is a viable and cost-effective alternative to the current hydrolysis procedure.

P-47   Determination of Chlorophenoxy and Other Herbicides in Food Commodities by QuEChERS and Detection by Liquid Chromatography with Tandem Mass Spectrometry

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Significant development effort has been expended in the pursuit of a method or series of methods to quantify the polar herbicide category of compounds. With reporting limits that continue to change downwards and an increasing number of herbicides in this category, our expansion of a validated method for the analysis of a single analyte to this broader range of contaminants was investigated. Gas chromatography with derivatization of the analytes was initially evaluated, employing trimethylsilyl diazomethane; however numerous derivatization byproducts impeded progress with GC analysis. Changing direction to LC/MS/MS allowed for easier sample workup, and inclusion of alternate cleanup steps. Starting from a very basic QuEChERS template, we evaluated extraction solvents, hydrolysis of herbicide esters, QuEChERS salt composition, post-extraction dilution schemes, and dispersive SPE cleanup steps. The ability to apply a

single method to vegetables, finished food products, grains, infant formula, and dietary supplements is an important consideration for an analytical lab. Data specific to the recoveries in select samples and instrument conditions will be presented.

P-48   Agricultural Screening of Volatile Organic Compounds as Indicators of Infestation by Portable Gas Chromatography

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The USDA APHIS Plant Protection and Quarantine (PPQ) mission is to "safeguard U.S. agriculture and natural resources against the entry, establishment, and spread of economically and environmentally significant pests." Currently agricultural inspections are primarily limited to visual inspections and concealed pests could escape detection. Developments in portable chemical technology have provided a means to use chemical signatures of concealed pests to detect their presence/absence during the inspection process. To facilitate deployment of innovative technologies through performance verification, PPQ investigated Electronic Sensor Technology (EST) 4300 Ultra-Fast GC Analyzer for detection of *Trogoderma granarium* (khapra beetle) infestation in rice. The primary objective of the verification test was to compare the performance of the field technology to laboratory-based measurements. Another objective was to evaluate the field technology's capability to discriminate between non-infested and infested rice. Due to quarantine restrictions associated with the khapra beetle, *Trogoderma variabile* (warehouse beetle) was selected as our surrogate. Methods were developed for the characterization of VOCs for rice using an Agilent 7890 GC with a 5975 MSD and a Gerstel Multipurpose Sampler. Methods were adapted to the EST 4300 Ultra-Fast GC Analyzer. Samples of non-infested and infested rice were prepared to assess independently the performance of the field technology.

P-49   Determination of endogenous concentrations of nitrites and nitrates in cheese: method development and validation using ion chromatography

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Nitrites and nitrates are nitrogen compounds that are naturally produced in the environment through reactions in the nitrogen cycle. Sources of nitrogen to the environment occur from fertilizer, livestock and sewage waste, precipitation, and fossil fuel combustion. As a result, dairy products produced from livestock can have endogenous concentrations of nitrates and nitrites obtained from uptake through grazing and the water supply. Nitrites and nitrates can also be intentionally added to cheese as a preservative. In the EU, up to 150 mg/kg of nitrates are allowed to be present in cheese. In the US, nitrites and nitrates are un-approved for use as food additives. In order to be able to investigate imported cheeses for nitrites and nitrates intentionally added as preservatives and the endogenous concentrations present in cheeses in the US marketplace, a method was developed and validated using ion chromatography with conductivity detection. A market sampling of cheese samples purchased in the Washington DC metro area was performed to determine endogenous concentrations. This technique was developed to be rapid and simple in both sample preparation and analysis by ion chromatography. Method validation and market sampling results are reported.

P-50   Detection of Pesticide Residues on Fruit Surfaces Using 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 immobilized on a quartz paper matrix that can be used to help identify four different pesticides: thiram, malathion, imidacloprid, and phosmet. In order to observe the desired Raman spectral signatures of these pesticides, apple skin contaminated with each chemical was swabbed with the SERS substrate followed by interrogation with 785 nm laser excitation. 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 2016 Code of Federal

Regulations for imidacloprid, thiram, malathion, and phosmet are 0.5, 7, 8, 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-51   Development of Analytical Method for Determination of Florpyrauxifen-benzyl and XDE-848 acid Using LC-MS/MS

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Florpyrauxifen-benzyl is a novel aryl picolinate herbicide developed in Korea by Dow AgroSciences Ltd. Chemical structure of aryl picolinate, well known as 6-Aps, can plant wither with a small amount by combining with certain auxin receptor of weed, so it is known that it has activation on post-emergence weed control. In 2017, the maximum residue limit (MRL) of florpyrauxifen-benzyl will be established for rice in Korea. According to MFDS (Ministry of food and drug safety, Republic of Korea) regulations, florpyrauxifen-benzyl residues will be defined as the sum of florpyrauxifen-benzyl and XDE-848 acid. Thus, a simultaneous analytical method is needed to estimate the residue levels of the parent compound and its metabolite. The objective of this study was to develop and validate on analytical method for the determination of florpyrauxifen-benzyl and metabolite (XDE-848 acid) in representative agricultural commodities. Samples were extracted with acetonitrile and partitioned with dichloromethane to remove the interfering substances, after purified through C_{18} SPE cartridge. The analytes were quantified and confirmed by liquid chromatograph-tandem mass spectrometer (LC-MS/MS) in positive-ion mode using multiple reaction monitoring (MRM). Matrix matched calibration curves were linear over the calibration ranges (0.005-0.5 µg/mL) for all the analytes into blank extract with coefficient of determination (r^2) > 0.99. For validation purposes, recovery studies will be carried out at three different concentration levels (LOQ, 10LOQ, and 50LOQ) performing five replicates at each level. The proposed analytical method will be used to as an official analytical method in Republic of Korea.

P-52   MRMHR: A Novel Scan Mode for Trace Level Pesticide Analysis of Cannabis Plant Extracts using High Resolution QTOF Mass Spectrometry

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State regulations for pesticides in cannabis products have a zero tolerance approach whereby any pesticide found that is not approved causes the material to be out of compliance which may result in removal of the product from distribution. Compliance with regulations for pesticides is most often performed using LC-MS/MS with a triple quadrupole mass spectrometer but the complex matrix of these samples often can make compound confirmation via ion ratios difficult. We have been investigating the use of a QTOF mass spectrometer using the MRMHR scan mode on a SCIEX X500R QTOF System to provide better confirmative data. With this scan mode it is possible to acquire MRM transitions at optimized collision energies which results in better sensitivity for quantitative applications. We are investigation two different aspects of this scan mode. One is to acquire data over a narrow range of masses which produces data similar to traditional QQQ data. The second is to set up two transitions at optimum collision energies for the fragments but one of the transitions acquires a full scan MS/MS spectrum that can be used simultaneously for quantitation and for compound identification. The results of these studies will be presented. The focus of the presentation will be to show quantitation data for the Oregon list of pesticides and to demonstrate the effectiveness of the MRMHR data for compound identification and target confirmation.

P-53   Development of Simultaneous Analytical Method for Pesticide Multiresidues in Chicken and Egg using LC-MS/MS and GC-MS/MS

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Chickens meat and eggs are the most consumed animal food in Korea. In chicken farms, pesticide treatment is a common practice for the elimination of parasitic insect such as mite. The purpose of this research is to develop a simultaneous

analytical method for pesticide multiresidues in chicken meat and eggs using highly selective and sensitive MS/MS technology (LC-MS/MS; Shimadzu LC-MS 8050 and GC-MS/MS; Shimadzu TQ 8040). Those pesticide of concern of residue (27 compounds) were selected and were classified into two groups of GC-MS/MS (9 compounds) and LC-MS/MS (18 compounds) depending on their chromatographic characteristics. Sample preparation was based on QuEChERS method (original and EN 15662) with some modification of extraction solvent (addition of formic acid). In the clean-up step, dispersive SPE containing MgSO4 and C18 was tested with those extract. Seven compounds were suitable for GC-MS/MS and 14 compounds were suitable for LC-MS/MS after sample treatment was optimized. In MS/MS, positive electrospray ionization was used with scheduled multiple reaction monitoring of transition ions. Method limits of quantitation were 5 ng/g and $r^2 > 0.99$ (linearity) was obtained with matrix matched standard for the most of compounds. Recovery test was carried out by spiking two levels (10 and 50 ng/g) to give 70-120% with relative standard deviation of ≤ 20% for the most of compounds. The method developed in this study could be used successfully for the monitoring of multiresidues of corresponding pesticide in chicken meat and eggs.

P-54   Proficiency-testing scheme using true samples for Gluten detection in processed food

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Food allergies represent a significant health problem in industrialized countries and the number of laboratories performing the analyses of allergens has gradually increased in recent years. The allergens detection in processed foods is a challenge for the laboratories: the extraction of denatured or altered proteins tends in fact to be difficult due to their reduced solubility as compared to native proteins. According to the requirements of ISO 17043 standard [1], laboratories participating in a proficiency testing scheme (PTS) must operate under routine conditions and analyze samples as close as possible to the real ones. To meet this requirement, BIPEA (http://www.bipea.org) set up a regular PT intended to the detection and quantification of gluten in cakes. The samples were made by preparing cakes including in the recipe wheat flour in well controlled proportions before the cooking process. Intrinsically contaminated cakes were obtained, closer to the reality than samples to which gluten is artificially added after the cooking process. The 20 laboratories participating in this test were required to return their results on a dedicated website after a period of one month, and a statistical treatment of the data was as usual performed by BIPEA according to ISO 13528. Assigned (consensus) values were calculated from the participants' results and the performances of the laboratories could then be evaluated individually and collectively according to ISO 17043.

P-55   How Heterogeneity in Treated Seed Pesticide Residues Provides Challenges for Measurement Quality Control

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Seed treatments are applied to seed surfaces to help seedlings battle insects and diseases and promote early season growth for crops. Treated seeds can have large and highly variable concentrations of pesticides, from units or tens of ppm, even within the same source of seeds. These seeds may be inadvertently present in final products for export, and may cause average concentrations of pesticides in these products to exceed maximum residue limits. The high variability of residues on treated seeds also provides challenges for measurement quality control. This study presents an LC/HRMS method for the analysis of 36 pesticides with quality control information for 8 pesticides. The variable application of pesticides to the seed surface made reliable spike and recovery experiments difficult to perform.   As a result, in-house wheat control samples were run for wheat alongside sample analyses and used for control charting. Relative standard deviations for sets of in-house control samples for 20 samples run over 6 months were as high as 50 % for prothioconazole. To further investigate, 100 individual seeds treated with Raxil Plus Shield were analyzed and relative standard deviations were found to range from 25 % for tebuconazole to 121 % for prothioconazole. This heterogeneity in seed concentrations is likely produced from the method of pesticide application. Strategies to assess and improve confidence in quality control procedures for the analysis of pesticide residues in treated seeds requires further attention.

P-56   Pesticides in wine: evaluation of QuEChERS method and dispersive liquid-liquid microextraction for the multiresidue determination by UHPLC-MS/MS

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Nowadays, there is an increasing concern about the health and safety impact of the widespread use of pesticides in agriculture including viticulture. Based on that, this work reports two multiresidue methods for the extraction of pesticides in wine. The QuEChERS method and the dispersive liquid-liquid microextraction using demulsified solvent (SDDLLME) were optimized for the determination of pesticides in red wine. Both methods were developed through evaluation of the main parameters affecting extraction efficiency. After the methods were validated and critically compared. The use of ultra-high performance liquid chromatography coupled to mass spectrometry (UHPLC-MS/MS) allowed to perform the determination with high selectivity and sensitivity. For the QuEChERS method, 97 compounds were validated in linear range of 0.5 and 50 μg L^{-1} with r^2 > 0.99. Limit of quantification of the method (LOQ) and determination (LOD) ranged from 10 to 20 μg L^{-1}. When SD-DLLME was used, 74 compounds were validated in the linear range of 0.1 and 2.5 μg L^{-1} with r^2 > 0.99. LOQ and LOD were between 0.1 and 0.2 μg L^{-1}. Both methods were applied for determination in real samples of wine. Pesticides residues of different classes were found in the samples in the concentration range of 0.8 to 55.3 μg L^{-1}. In view of the results obtained, both methods showed low reagents consumption and waste generation. Besides, the use of SD-DLLME proved to be a viable alternative to the QuEChERS method for the multiresidue determination of pesticides in wine.

P-57   Evaluation of a new automated QuEChERS sample preparation technique

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The Florida Department of Agriculture, Chemical Residue Laboratory has the capability to detect and accurately quantify hundreds of chemicals in Florida's fresh produce samples; therefore, it's important to have repeatability and consistency when extracting pesticide residues. The performance of a new automated sample preparation system was evaluated in comparison to the current manual sample preparation technique, using modified QuEChERS (Quick, Easy, Cheap, Effective, Rugged, Safe) methodology. The Teledyne Tekmar AutoMate Q40 is a system specifically designed and optimized to automate the QuEChERS workflow; reducing human errors and improving precision and accuracy. A variety of fresh fruits, vegetables, and honey samples were extracted manually and using the AutoMate Q40, then analyzed by GC/MS and LC/MS. Precision of the AutoMate Q40 was evaluated by extracting samples in triplicate; 70% of the samples were under 0.02 ppm difference between the highest and lowest recovered concentration, and the other 30% was due to the analysis of poor performing compounds, like captan. The two different extraction techniques were compared against each other by looking at the recovery of incurred pesticide residues; 85% of the samples were within a 25% difference between the manual extraction and the AutoMate Q40 extraction. Further research must be done on incurred residues and AutoMate Q40 method variations to yield higher recoveries.

P-58   GC-MS/MS Survey of Pesticides in Botanicals Using a Modified QuEChERS Method

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Sales of herbal and botanical dietary supplements have increased every year for over a decade in the United States. An internet search for "purchasing herbal and botanical supplements" delivered over 2 million results.   Herbal and botanical plants are valuable agricultural crops, and pesticides may be used to protect them from damage, preventing economic losses. The Forensic Chemistry Center (FCC) receives numerous samples that contain, or are declared to contain, herbal/botanical materials. An FCC method developed for the analysis of pesticide residues in cigarette tobacco was applied to various herbal/botanical supplements and bulk materials. For this study, GC-MS/MS with multiple reaction monitoring (MRM) was utilized for the identification and determination of 56 pesticides in the samples chosen. The GC-MS/MS procedure compliments LC-MS/MS methods by providing critical coverage for most non-polar pesticides that are not amenable to electrospray ionization (ESI) in the positive mode. Validation experiments were carried out by applying the procedure to a representative organic botanical product, which was also used to prepare matrix matched standards. A survey of the types and amounts of pesticide residues found in various botanical products will be presented, including notable matrices such as herbal cigarettes, Kratom, Damiana, grape seed extract and olive leaf extract.

P-59 Evaluation of Various Dispersive Cleanup Sorbents and Their Effect on Pesticide Recovery in Cannabis

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Cannabis flower is a very complex matrix which makes the analysis of pesticides quite challenging. The complexity of the cannabis matrix is shown in the unbalance between cannabinoid content and pesticide level. The cannabinoids are produced at 10-20% levels or greater which correlates to 100,000-200,000 ppm whereas the pesticides even at 100 ppb is only 1.0×10^{-5} %. Other compounds like terpenes and lipids produced at 0.001-0.5% (10-5000 ppm) can also cause issues with mass spectrometers as they move through the system depositing on the flow path of the instrument. Many sample preparation approaches have been used from simple dilution, QuEChERS methodology through to solid phase extraction. Removing enough matrix to analyze the pesticides and reducing the deleterious effects on the system is crucial. This study took a practical look at the effect sorbents and sorbent mixes had on matrix removal and pesticide recoveries in a dispersive SPE (dSPE) format. One gram of homogenized cannabis material was extracted with acetonitrile. The extract was subjected to a systematic approach where sorbents were incrementally increased and various sorbent mixtures were formulated. This study is a guide to better understand the effect various sorbents have on pesticide recovery in cannabis flower. Criteria for acceptable pesticide recoveries are 70-120% and S/N ≥ 10. Pesticide recovery for the various dSPE formulations were evaluated by triple quadrupole LC/MS using a superficially porous column and on-line dilution.

P-60 A Novel Approach to Sensitive Pesticides Analysis in Cannabis by GS/MS/MS

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The challenge of quantifying pesticide residues in Cannabis flower is a complex problem that is due in part to the great disparity between concentration levels of naturally-occurring cannabinoids and incurred pesticide residues, as well as the high terpene content of the plant. The typical extraction process gives rise to the potential for low pesticide recoveries and deleterious effects on the analytical instrumentation caused by co-extracted material. We present an approach to sample preparation that includes extraction of 1g homogenized material with acetonitrile followed by cartridge SPE, dilution of eluate with a less polar solvent mixture which is then subjected to dispersive SPE, and a final dilution step. GC/MS/MS analysis is performed on a system equipped with mid-point column backflush using GC columns of differing phase polarities and a highly efficient ion source. The method utilizes the external standard technique. In addition to highly diluting the sample extract, the addition of small molecule analyte protectants prior to injection was evaluated. The calibration range was 0.2 – 20 ng/mL (concentration in autosampler vial). Pesticide recoveries were acceptable (70-120%) for the list of over 70 targets, and LOQs in Cannabis were determined to be 0.1 mg/kg for the 85% of the target list. Performance of the method with non-identifiable, real samples is presented.

P-61 Analysis of Cannabis for Pesticide Residues by GC/Q-TOF

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As of the November 2016 election, 29 states have approved the use of medical cannabis and eight states, representing 65 million people, have approved recreational use by adults. Canada allows medical use and is on a path to full legalization. Because the US government still classifies cannabis as a schedule 1 drug, all legislation controlling the growing, testing and use of cannabis products is done at the state level. There is no uniformity in the regulations and their enforcement. Pesticide use on cannabis plants is very controversial, but is loosely regulated in most states. It is clear that pesticide residue testing is important for a product that may be eaten or inhaled. This paper describes the analysis of cannabis extracts for pesticide residues using a accurate mass high resolution GC/Q-TOF. Chromatograms

are analyzed by applying a "find by fragments" approach using a personal compound database and library (PCDL) containing about 852 exact mass spectra of pesticides and other contaminants. The software screens for all compounds in the PCDL in just a couple of minutes. Standards are only needed for those pesticides that are found and need to be quantified. This approach has also been used to identify pesticide residues in food and herbal extracts. Sixteen samples of confiscated marijuana were analyzed by this method. Twenty-one pesticides, two organophosphorus fire retardants and three PAHs were tentatively identified in the samples.

P-62   Polycyclic Aromatic Hydrocarbon (PAH) evaluation in complex food matrix using Triple Quadrupole Gas Chromatography Mass Spectrometry (GC-MS/MS)

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Polycyclic aromatic hydrocarbons (PAHs) are formed from the incomplete combustion of organic matter. In food, PAHs are generated during the curing and processing of raw foods or meats cooked over an open flame. Trace levels of PAHs are closely monitored in food as diet is the major source of exposure to PAHs. We introduce an optimized extraction procedure to investigate PAHs in fatty and complex matrix. The challenge of analyzing PAHs is due to their chemical properties. PAHs are resistant to chemical reactions, accumulate rather than degrade, and tend to de-sublime making PAHs difficult to vaporize. United States and European Union-regulated PAHs were evaluated using a modified Gas Chromatograph Electron-Impact Triple Quadrupole Mass spectrometry (GC-EI/MS/MS). A self-cleaning ion source and post-run mid-column backflush allowed the source to be kept clean from PAH and sample deposition.

P-63   Multi-Residue Pesticides Screen and Quantitation in Food Matrices Using HPLC and Newly Developed Triple Quadrupole Mass Spectrometer

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Pesticides are vital to the success of crop production. By design, pesticides can be toxic to humans if consumed in harmful doses; therefore, regulatory agencies have set maximum residue levels (MRL) for hundreds of pesticides and their metabolites in foods. Most MRLs are set at low ppb levels, posing significant challenges to screen and quantify hundreds of analytes in complex food matrices simultaneously. In this presentation, we demonstrate a screening and quantitation method for 250 pesticides and their metabolites using HPLC and a newly developed triple quadrupole mass spectrometer. The new instrument features robust performance and easy maintenance. Orange, avocado, and black tea matrices were chosen to represent most fruits, vegetables, and dried herbs. Matrices were extracted using Agilent QuEChERS kits. A new EMR-Lipid kit was used for avocado to remove excess amounts of fat. Most analytes could be detected at 1ng/g in orange and avocado and 5ng/g in black tea, highlighting the excellent sensitivity of the new instrument. The chromatographic method consisted of using 3.0 x 150mm, 1.8um C-18 column, with 4.5mM ammonium formate, 0.5mM ammonium fluoride, and 0.1% formic acid as additives in water-methanol mobile phases. A dynamic MRM method was employed to have optimal dwell time for analytes in a narrow time window, with 2-4 MRMs per analyte. This could be accomplished due to retention time stability provided by the high-quality LC system and analytical column. Most of the pesticides could be accurately quantified far below MRLs with high precision (%RSD < 20%).

P-64   Analysis of Mycotoxins in Food Matrices Using HPLC and an Innovative Triple Quadrupole Mass Spectrometer

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Mycotoxins can be found in fungus-contaminated crops and are toxic to humans and animals; therefore, mycotoxin levels are monitored in foods to minimize the risk of ingestion. Mycotoxins need to be quantified in various food matrices at levels below the currently set Maximum Residue Limits (MRL) by regulatory agencies. Maize, peanut, and black pepper were chosen as matrices because they are all regulated for mycotoxins, and they represent grains, nuts, and spices. The mycotoxins were extracted using Agilent QuEChERS kits best suited to each matrix, in combination with a novel modified lipid removal sorbent. A fast, robust, and precise method was developed for the detection of 12 commonly regulated mycotoxins, using an innovative triple-quadrupole mass spectrometer (QQQ). The targeted mycotoxins were separated

using a 3.0 x 150mm, 1.8mm, C18 reversed-phase column, with mobile phases consisting of water and methanol, each modified with 0.1% formic acid, 5mM ammonium formate, and 0.5mM ammonium fluoride. Instrument detection limits (IDL) for analytes were below the MRL in the studied, highlighting the excellent sensitivity of the new instrument. Two MRM transitions were monitored in a dynamic fashion for optimal dwell time for each compound. Excellent method repeatability demonstrated the robustness of the system, relative standard deviations (RSD%) of <20% at lowest level of quantitation. The combination of the clean-up of matrix interferences, chromatography, and the newly developed QQQ allows for the sensitive and robust detection of mycotoxins.

P-65 Method Performance of Food and Water Samples for Glyphosate and its Metabolite by LC/MS/MS

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Glyphosate is the active ingredient in the popular herbicide Roundup® and is used throughout the world. Glyphosate is a broad-spectrum systemic herbicide. It is an organophosphorus compound, specifically a phosphonate. It kills weeds, especially annual broadleaf weeds and grasses that compete with crops. It inhibits a plant enzyme involved in the synthesis of three aromatic amino acids: tyrosine, tryptophan, and phenylalanine. Therefore, it is effective only on actively growing plants. 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. Reliable sample preparation and analysis is needed to routinely analyze glyphosate and its metabolite, aminomethylphosphonic acid (AMPA). However, glyphosate and its metabolite's high polarity can be quite challenging not only extraction from food but in its analysis. Using various food samples, we examine extraction and cleanup procedures including the QuEChERS for polar pesticide (QuPPe), direct water extraction, and a solid phase extraction cleanup.   This poster will present the results of this evaluation and the performance of a direct LC/MS/MS method of analysis. Matrix effects, repeatability and sensitivity will be described.

P-66 LC/MS/MS of Glyphosate and AMPA: Comparison of Direct Analysis and FMOC Derivatization

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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 compare the use of derivatization of glyphosate and its metabolite, aminomethylphosphonic acid (AMPA), in both types of samples to direct analysis with no derivatization. The FMOC derivatization provides a detection limit of 0.1 ppb while direct analysis is 1.0 ppb. However, the derivatization produces a sample that can foul the instrument and the derivative must be analyzed within 24 hours due to its degradation. In contrast the direct analysis of the two polar compounds requires much less sample preparation, is relatively clean and is stable for at least 2 weeks.   The FMOC derivatization employees a reversed-phase C18 chromatographic separation using ammonium acetate in water and acetonitrile with triple quadrupole MRM detection. The direct analysis uses a unique ion-exchange column, the Metrohm Asupp 4, 4.0x200 mm, to effect an excellent separation of the analytes and other polar pesticides. Details of the sample preparation, the analytical methodologies and their results will be described.

P-67 Advantages of Reversed Sandwich Injection for Pesticide Residue Analysis

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The use of matrix-matched calibration standards has always been widely accepted in pesticide residue analysis to ensure accurate and reliable quantitation results in different commodities. This is important because pesticide response is influenced by the various matrices, and may lead to biased quantitative results. Performing matrix-matched calibrations eliminates the response biases, and allows for more accurate identification and quantitation results. However, the preparation of matrix-matched calibration standards can be a tedious and time-consuming procedure, especially when multiple sample matrices are analyzed. This practice also introduces the possibility of human errors during the

preparation, affecting the analytical results. The Agilent 7693A ALS system's Reversed 3-Layer Switch sandwich injection functionality allows for the trouble-free addition of the internal standard to the calibration standards, and simultaneous injection for each of four different food commodities to simplify the creation of matrix-matched calibration standards. Over 50 target selected pesticides were selected and compared throughout the different matrices. While maintaining the matrix as the bottom layer in the 3-layer sandwich injection, over 85 % of the target pesticides achieved a calibration curve with an R2 ≥ 0.991 (1.25 ppb to 62.5 ppb). All analyzed pesticides obtained a %RSD for repeated measurements at 1.25 ppb of ≤ 30 %, and 85 % of the analyzed pesticides were found to have a Limit of Quantitation (LOQ) ≤ 0.1 ppb.

P-68   Reducing Run Time and Solvent Usage with EPA Method 3640A (GPC Cleanup) Using a New Column Packing Material

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EPA Method 3640A for Gel-Permeation Chromatography (GPC) cleanup uses 70g of SX3 styrene-divinylbenzene Biobeads and requires 65 minutes of run time using DCM as a mobile phase. The maximum lipid loading capacity for this traditional column is 1 gram. With the introduction of the J2 Scientific Express™ column, run times were shortened to 40 minutes with a corresponding reduction in solvent usage. However, the maximum lipid loading for the column was also reduced to 500mg. A propriety stationary phase has been developed that reduces the run time for both the 1 gram and 500mg lipid-loading capacity columns while maintaining the EPA method's resolution and analyte recovery requirements. Comparisons between the Express™ column and the new columns are presented. Dual column chromatography, using traditional and Express™ columns, are also presented where up to 2 grams of lipid can be injected in a single run saving time and solvent.

P-69   Automation of EPA Method 525.2 – Determination of Organic Compounds in Drinking Water

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EPA Method 525.2 is used to determine a wide array of organic compounds in raw and finished drinking waters. One (1) liter water samples are passed through chemically bonded C18 solid phase extraction (SPE) cartridges or disks. The analytes of interest are recovered with ethyl acetate and dichloromethane bottle washes which are used as elution solvent for analyte recovery from the dried sorbent bed. Residual water is removed with subsequent sodium sulfate drying of the elution solvent prior to concentration and analysis by gas chromatography/mass spectroscopy (GC/MS). The PrepLinc™ Large-Volume Injection (LVi) system allows the user to pass large volumes of aqueous sample matrix through commercially available SPE cartridges and disks. The autosampler will accommodate 1 liter sample jars, therefore sample is taken directly from the container used to collect the water in the field. The sample jar rinses are used to elute the analytes of interest to collection tubes, or to an AccuVap module for concentration directly to autosampler vials. 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 labor intensive process of manually loading 1 liter of water to an SPE, subsequent elution, and concentration for analysis. An evaluation of several commercially available C18 SPE cartridges and disks was performed for comparison to the J2 Scientific 525.2 SPE cartridge.

P-70   Improved GC/MS Analysis of Pesticides Through Inhibition of Acid-Base Chemistry in Solvents

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Development of GC/MS analytical methods for pesticides has revealed a significant number of compounds susceptible to acid-base reactions in commonly used solvents. Pesticides containing carbamate functional groups constituted the majority of those identified. Collected data revealed formation of alcohol, **2** from carbamate pesticide, **1**. In addition several pesticides with stereocenters or highly reactive carbonyl groups were observed to form various reaction products in neutral solvents. Addition of formic or acetic acid has inhibited these reactions, increased instrument response and resulted in greater consistency in GC/MS analytical results.

P-71   Analysis of L-Theanine in tea by HPLC with post-column derivatization.

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Theanine is a neurologically active amino acid found in tea plants. L-Theanine is a dominant amino acid in green tea and is responsible for its unique pleasant taste as well as known relaxation effect. Theanine also has been proven to reduce physical and mental stress, and improve cognition and mood.
Cation-exchange chromatography using post-column Ninhydrin reagent Trione® and UV detection has shown unmatched reproducibility and selectivity in the analysis of free amino acids in complex matrices. The Pinnacle PCX post-column derivatization system allows for shortened run times when utilizing column temperature gradients.
We introduce a simple and robust method for the analysis of L-Theanine in addition to the other free amino acids in tea leaves.

P-72   Analysis of Cannabinoids using HPLC with post-column derivatization.

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Broader acceptance of medical cannabis use increases the need for analytical methods capable of determining the active compounds of cannabis. A new HPLC method with post-column derivatization was developed to analyze cannabinoids in cannabis plants as well as in cannabis-containing edible products. This post-column method is based on reaction with Fast Blue Salt reagent, a well-known color-forming reaction that is used in drug tests to detect cannabinoids via test-tube and thin-layer chromatography. Detection at 475 nm is performed using a UV/Vis detector.
Our method implements a simple extraction with acidified water/acetonitrile followed by QUECHERS sample clean-up. The same procedure is applicable to both plant materials and edible products containing cannabis. The method is suitable for analysis of the major neutral cannabinoids such as THC, CBD and CBG as well cannabinoid acid (THCA) with high sensitivity and selectivity of detection.

P-73   Integration of sample cleanup and enrichment with chromatographic analysis

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Integration of solid phase extraction or column cleanup with chromatographic analysis may take two approaches: 1) Direct coupling, in which a sample loaded in a cleanup column is completely transferred to the analytical column using a switching valve; 2) Indirect coupling, in which samples are treated like conventional offline cleanup. A portion of the collected fraction is then injected into the analytical column using a built in auto sampler or by triggering the auto sampler from the HPLC. The direct approach can give a much higher sensitivity than indirect approach,   whereas the indirect approach is easier to find suitable cleanup columns and faster for method development. One of the major challenges in a direct approach is finding suitable cleanup columns that can give effective cleanup and allow use of compatible mobile phase. If one cleanup column cannot process at least 50 samples, an expensive device for loading cleanup columns need to be used   which makes this type of online SPE device much more expensive than those based on indirect approach.   This presentation will show how to avoid peak dispersion and prolong life span of a cleanup column in direct online SPE using analysis of PAHs in water as an example. We will also show how to increase sensitivity in indirect online SPE. Finally we will demonstrate how to combine these two online approaches into one device so that the disadvantages can be avoided.

P-74   High Sensitive and High Throughput Analysis of Pesticide Residues in Botanical Ingredients using GC-MS/MS

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Use of dietary supplements is increasing in the United States. These dietary supplements are made from various dried

botanicals, and residual pesticides in them have to be monitored to ensure their quality and prevent exposure. However, for very low amounts of pesticides, gas chromatography requires a slow oven program to obtain enough dwell time when coupling with a mass spectrometer. In addition, many botanical ingredients are dry and have high interference. These two challenges make it difficult to build a highly efficient and sensitive analysis. In this study, ultra-fast mass spectrometry (UFMS) was used to increase dwell time, enhance sensitivity, and throughput by coupling it with a modified QuEChERS method using solid phase extraction (SPE). The UFMS achieves greater than 800 MRM transitions per second and this technology achieves enough data points to obtain good reproducibility even while using a faster GC program. Also, this can maximize dwell time in fast multi component analysis. The modified QuEChERS and SPE method ensured high quality data by reducing matrix interferences. This analytical method enhanced throughput by 65% comparing with standard analytical condition and covered a wide calibration window ranging from 1 to 1000 ng/mL. Also, it achieved acceptable mean recoveries of 57 to 104 and 48 to 101%, and high accuracies, lower than 13.7 and 12.6%RSD in 50 ng/g fortified green tea and chamomile samples for 18 representative pesticides.

P-75   Bitter Medicine: Pesticides in Cannabis by modified QuEChERS and GC-MS-MS

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Pesticides have been detected in cannabis offered for retail sale in many states, including Colorado, Oregon, and Washington. Although crop protection agents are an important part of agricultural production, such pesticides may be disallowed in cannabis for regulatory reasons. LCMS analysis has shown widespread use of these substances, however to detect the complete range of chemical residues which may be present, GCMS is required. Previous GCMS analysis resulted in high background due to the high THC levels as well as rapid instrument fouling with such dirty samples. Therefore we developed a GC-MS-MS analysis using a modified QuEChERS extraction and dispersive SPE cleanup which reduces cannabinoid interferences and prevents premature instrument fouling. We applied our method to detection of pesticides in a variety of dried cannabis flower samples and cannabis concentrates. This new method extends the capability of pesticide detection in cannabis to substances requiring GC-MS-MS detection with a rapid, sensitive, and selective analysis.

P-76   Analysis of Cannabis & Hemp Products for Heavy Metals

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The cannabis industry has taken the world by storm and has flooded the market with new products. Recently, concerns have arisen around the safety of this largely unregulated market. Cannabis testing laboratories emerged to fill the need for specialized testing for cannabinoid potency, pesticides, bacteria/mold and other potential contaminants. Sadly, a significant group of contaminants has been largely ignored: toxic metals.
Recreational cannabis and hemp are both the same species. Federally legal hemp products are easy to obtain by the general public. Hemp products are also used as base for cannabis products & cannabinoid extracts. However, due to a ban on hemp cultivation in the US, virtually all of the hemp used in the US is imported from China, India, Eastern Europe, and Canada. Studies of other commodities exported from these countries have reported widespread heavy metal contamination (i.e. spices, teas, grains etc.).
Cannabis plants are potential bio-accumulators of heavy metals. In the production of these products, a large amount of plant material is processed to extract concentrates and oils, thereby increasing the risk of heavy metal contamination. The scope of this study was to analyze various legal hemp products currently on the market for heavy metal contamination and use hemp as a model for methods development for restricted products. Samples were digested using microwave digestion and analyzed by ICP-OES and ICP-MS.

P-77   Analysis of 648 Pesticides in Foods by LC-MS/MS Utilizing the Raptor Biphenyl Column

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There are more than 1,000 pesticides used globally on soil and crops. With the ever-increasing international trade within the food industry, regulatory bodies around the world have increased the number of regulated pesticides and the maximum residue levels (MRLs) allowed in food commodities. National pesticide monitoring programs create new

challenges for food safety laboratories as the number of pesticides required for analysis is increasing together with an expanded range of food products. In this poster we present the development of an LC-MS/MS method using a Raptor Biphenyl LC column for screening and quantifying over 648 pesticides in a single analysis. To evaluate the method, QuEChERS extracts of mint, tomato and apple were provided by a commercial laboratory as raw acetonitrile extracts and spiked with 648 pesticides prior to analysis. The method was evaluated in matrix to ensure that the reporting limits were in agreement with recognized MRL's.

P-78   Preparation, UHPLC-MS/MS Analysis, and Accurate Quantitation of Nitrofurans Metabolites, Chloramphenicol, and Florfenicol in Seafood

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We developed and validated a method for the extraction, identification, and quantitation of the four most important nitrofuran metabolites (3-amino-2-oxazolidinone (AOZ), 3-amino-5-morpholinomethyl-2-oxazolidinone (AMOZ), semicarbazide (SC) and 1-aminohydantoin (AHD)) and two phenicols (chloramphenicol and florfenicol) in a variety of seafood commodities. Samples were derivatized with 2-nitrobenzaldehyde in acidic conditions overnight and subsequently extracted and analyzed by liquid/liquid extraction techniques in conjunction with UHPLC-MS/MS. Two product ion transitions per analyte were required for identification and confirmation which contributes to a high degree of selectivity. Quantitation has been performed using commercially available derivatized nitrofurans metabolites and isotopically-labelled internal standards using in-solvent matched calibration curves. Recoveries were found to be 90-100% at fortification levels of 0.25, 1, 5 and 10 ng/g for nitrofurans and 0.1, 0.25, 0.5, and 1 ng/g for phenicols. The limit of detection (LOD) was found to be 0.25 ng/g for AMOZ and AOZ, 1 ng/g for AHD and SC, and 0.1 ng/g for phenicols, when 2 g of seafood were extracted. Various extraction methods, standards stability, derivatization efficiency, and improvement of the conventional quantitation techniques have also been discussed.

P-79   Seafood Safety: Preparation of Seafood Samples for Dyes Analysis

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Increased globalization of the food supply and the rising demand for seafood has raised food safety concerns. Regulatory agencies are tasked with ensuring seafood is free of contaminates and safe for consumers. Triphenylmethane dyes are used in aquaculture production to treat seafood. The dyes are readily available, inexpensive, and effective. Due to the toxicity and mutagenicity of triphenylmethane dyes, the U. S along with other countries have prohibited it for the treatment of fish to be used for human consumption. This poster presents the Florida Department of Agriculture and Consumer Services (FDACS), Chemical Residue Laboratories (CRL) preemptive stance of processing seafood samples for triphenylmethane dyes. Sample processing is an important step and the chances of introducing error are highly likely. Some of the common processing errors associated with subsampling in the laboratory include: splitting, mass reduction, grinding and blending. CRL decided to combat these errors by freezing samples and selecting the proper tools to process the entire laboratory sample. A 15-quart commercial food processor was purchased to comminute an entire sample weighing approximately 6 lbs (~ 2.7 kg). The comminuting technique presented produces a better representation of the decision unit, offers better heterogeneity, and ensures defensible samples.

P-80   Stone Crabs; Heavy Metal Contamination and Risk Assessment

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Stone crab samples were collected from processing outlets and retail outlets in Florida in 2011-2012 and 2016- 2017. Samples were analyzed for cadmium, lead and mercury by ICP MS after closed vessel microwave extraction. Results ranged from 0.07 µg/g - 0.26 µg/g for mercury. There were no quantifiable levels of cadmium or lead detected. The weight of the claw and claw lengths were measured to determine if they were correlated to contamination levels. Stone

crab claws are good sources of lean protein. A single serving (114 g, ~ 3 medium claws) contains, 17.6 g of protein, no fat, 53 mg of cholesterol and 353 g of sodium. Stone crab claws contain no omega 3 fatty acids.

P-81 Strategies for Extraction and Purification of Tetrodotoxin and Saxitoxin from Fish Filets with LC-MRM-MS Analysis

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Tetrodotoxin (TTX) and Saxitoxin (STX) are small, non-protein, alkaloid toxins commonly associated with puffer fish and shellfish poisoning events. These toxins can co-occur domestically in marine and freshwater fish and produce similar symptoms in affected people. A fast and sensitive detection method is required to facilitate a rapid response to TTX- and STX-related illnesses. STX is regulated in shellfish with a maximum limit of 0.8 mg/kg tissue. TTX is not regulated, although it has a similar potency to STX and elicits safety concerns at equivalent concentrations. The presence of TTX in a sample is an additional concern because often those fish have been imported illegally. Our goal is to establish a detection method that will unequivocally identify TTX or STX at least 10x below the anticipated safety level, with a robust sample preparation protocol for different types of fish products. This presentation will discuss the development of an optimized method for extraction of TTX and STX toxins from fish followed by detection and quantification using liquid chromatography-tandem mass spectrometry (LC-MS/MS). The extraction of TTX, STX, and their analogues has been optimized using incurred fresh/frozen fish and salt-preserved fish and verified using naturally-contaminated samples. Extraction of toxins from fish matrices has presented a difficult challenge due to high levels of salt, lipids, and collagen and other proteins present in the samples and the highly polar nature of the toxins. Discussion of sample preparation approaches for concentration and purification of TTX, STX, and their related metabolites extracted from fish will be presented.

P-82 Triphenylmethane Dye Residue Analysis in a Variety of Aquacultured Seafood Matrices including Highly Processed Canned Foods

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AOAC Official Method of Analysis 2012.25 is for the LC-MS/MS determination of residues of three triphenylmethane dyes that are commonly used illegally in aquaculture (malachite green, crystal violet, brilliant green) and their metabolites (leucomalachite green and leucocrystal violet). This method has been tested and validated for use in trout, salmon, catfish, tilapia, and shrimp. Additional studies of matrix effects for pangasius and boiled shrimp matrix have also been reported. In order to test the applicability of AOAC Official Method of Analysis 2012.25 on a wider scope of regulated aquaculture products, residue extraction studies were conducted on additional types of raw fish matrix (Arctic char, barramundi, hybrid striped bass, pompano, and seabream), scallops, frog legs, and eel. Highly processed canned eel and dace products were also analyzed. The processed eel and dace products selected for testing were typically packed in oil and contained salt, sugar, flavorings, spices, sauces, and/or preservatives. AOAC Method 2012.25 performed well in these matrices with very little difference noted in method performance compared to the matrices tested in the original validation studies, regardless of the type of matrix and presence of additional food ingredients. The dyes and metabolites were recovered from the various seafood matrices with greater than 80% accuracy and less than 15% RSD. The method detection level for the five analytes was below 0.5 mg/kg (ppb).

P-83 Pesticide Multi-Residue Analysis in Red Swamp Crayfish Using QuEChERS and HPLC-MS/MS

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Crayfish, also known as crawfish, crawdads, or freshwater lobsters, are crustaceans cultivated and consumed worldwide. Crayfish are raised in fresh water, but also reside in paddy field environments potentially exposed to pesticides used on rice or other aquatic plants. In this study, a multi-residue analytical method using QuEChERS sample preparation followed

by high performance liquid chromatography–tandem mass spectrometry (HPLC-MS/MS) analysis was developed for rapid determination of 60 pesticides, including 16 insecticides, 23 fungicides, and 21 herbicides, in whole crayfish and crayfish meat. Extraction solvents, partitioning salts, and dispersive solid phase extraction (d-SPE) cleanup sorbents were optimized with respect to analytes' recoveries and RSDs. The developed method is based on QuEChERS extraction of 10 g crayfish with 10 mL MeCN, partitioning with 3 g NaCl, and d-SPE cleanup with primary secondary amine (PSA). Satisfactory recoveries of 70-120% and RSDs <20% at 3 spiking levels were achieved for 50 pesticides in both whole crayfish and crayfish meat samples. Different HPLC conditions (isocratic and gradient elution) were tested and optimized to increase analytes' responses, reduce matrix effects, and achieve better ruggedness. The limit of detection was 1-10 ng/g in whole crayfish and 5-10 ng/g in crayfish meat. The developed method is quick, easy and reliable for multi-residue pesticide analysis in red swamp crayfish. To test the developed method on real-world samples, crayfish products were sampled from various seafood markets in China, and results will be presented.

P-84   Use of Chemometrics to Identify Marker Compounds

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Chemometrics is nowadays finding applications in a wide range of disciplines to improve analysis of compounds. This presentation aims to demonstrate how chemometrics based multivariate analysis is able to extract distinctive information from chemical data by using mathematical and statistical methods.
Extracts of basil plants cultivated by organic and conventional farming practices were tested with gas chromatography/mass spectrometry (GC/MS). Baseline correction and retention time alignment were performed prior to data processing. Partial least squares discriminant analysis (PLS-DA) was used to build classification models. Bootstrapped t-statistical weight feature selection method was used to select informative features (marker compounds) from GC/MS chemical profiles of organic and conventional basil extracts. Characteristic components in basil extracts were putatively identified. For method validation, selected maker compounds were used to construct economic classification models, e-PLS-DA. Both the PLS-DA and the e-PLS-DA classifiers were used in parallel to classify a new validation set collected 2.5 months later with no parametric change to experimental procedure. Classification accuracy of e-PLS-DA and PLS-DA classifiers was compared. Results showed that marker compounds have been successfully identified from the gas GC/MS data of basil extracts.
Feature selection proves valuable as a powerful and cost-effective approach for identifying marker compounds in chemical data. As all algorithms can be implemented in any numerical computing environment such as MATLAB, C, C++, Python, and R languages, expensive specialized data processing software is unnecessary.

P-85   Florida's Pesticide Residue Regulatory Program – FY 16 - 17

Jo Marie Cook

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Each fiscal year, from July to June, Florida's Chemical Residue Laboratory screens hundreds of fresh fruits, vegetables and honey for pesticide residues. The multi-residue screen has been expanded to over 500 analytes using Orbitrap for LC/MS. Florida's pesticide regulatory program focuses on Florida grown products and commodities that have been found violative in the past. In addition, the laboratory has been validating methods for the analysis of veterinary drugs in seafood. The poster will summarize analytical findings and detail violations by commodity and pesticide. Commodities with the most pesticides in a single sample and the frequency of pesticides by commodity will be shown. Overall, the incidence of violations remains very low, even in a program designed to target problems.

P-86   Ensuring your pet's health safety: Quantification of illegal, multi-class antibiotics in pet food and treats by LC-MS/MS

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Use of veterinary antibiotics in agriculture for disease prevention and growth promotion can result into presence of antibiotics in animal tissues. Antibiotics used in animals can be transferred to pet food/treats during the pet food production. Pet food/treats contaminated with antibiotics have been linked with vomiting, diarrhea, increased urination,

decreased appetite and antimicrobial resistance in dogs. Recent cases of dog illnesses have alarmed the pet owners and created a need to develop tools to determine the presence of a wide range of antibiotics in pet food/treats. Typically, analysis of antibiotics in pet food involves extraction of antibiotics with acetonitrile followed by liquid chromatography/ tandem mass spectrometry (LC-MS/MS). However, this approach lacks effective cleanup of matrix and may induce signal suppression of analytes during mass spectrometric detection. In current work, we aimed to develop an effective strategy to extract multiple classes of antibiotics from pet food/treats combined with simple pass through solid phase extraction (SPE) clean up. In first step, antibiotics were extracted using an extraction solvent which contained phosphate buffer and acidified acetonitrile. Next step involved pass through cleanup of extracts using SPE column. Samples were analyzed by liquid chromatography-triple quadrupole mass spectrometry equipped with electrospray ionization source. Current approach successfully analyzed seven different classes of antibiotics classes. Acceptable recoveries were obtained ranging from 70-120% and <20% RSD for majority of analytes. Current approach allowed detection and quantification of multi-class antibiotics in pet food/treats and the quick SPE cleanup avoided the possibility of instrument contamination due to matrix.

P-87　Analysis of Peptide Antibiotics in Milk using High Resolution Mass Spectrometry

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Polypeptide antibiotics, such as colistin, polymyxin, and bacitracin, have been historically used in food-producing animals to improve their growth conditions and for the disease prevention. These peptide antibiotics are not well incorporated into current regulatory screening because simultaneous determination of multiple peptide antibiotics is relatively difficult due to the difference in their physicochemical properties. Recently, drug-resistant bacteria have emerged toward some of these antibiotics, particularly colistins which are now re-considered as last-resort for human clinical treatment. Therefore, the presence of these peptide therapeutics in human food or animal feed should be assessed and incorporated into the current surveillance program. The aim of this study is to develop a simple and rapid analysis method to screen a wide range of residual peptide antibiotics in milk based on liquid chromatography-high resolution mass spectrometry (LC-HRMS). A quadrupole time-of-flight (Q-TOF) instrument is used and the precursor ions (combination of MH^+, MH_2^{2+}, MH_3^{3+} ions) are integrated for quantification based on their exact mass measurement followed by tandem MS (MS/MS) for confirmatory product ions. Milk samples are extracted with acetonitrile containing 0.3% (v/v) formic acid and 0.06% (v/v) trifluoroacetic acid under sonication. The samples are centrifuged and passed through 0.2um filter prior to mass spectrometry analysis. In this method, we are able to determine the commonly used peptide antibiotics including colistins, polymyxins, bacitracin A, enramycin A, enramycin B, virginiamycin M1, virginiamycin S1, and novobiocin in the milk. The overall recovery for each compound in the preliminary results is within 80-110% (RSD=9-18%) after correcting for matrix effects.

P-89　Evaluation of sample preparation techniques for the extraction of multiclass veterinary drugs residues in Salmon (salmo salar) by UHPLC-MS/MS

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Worldwide growth of aquaculture in the last decades is evident and it is a strategic development field. Often, intensive and semi-intensive use of antibiotics prevents the emergence and spread of infectious diseases in fish. The greatest concern on antibiotics residues is the development of pathogens that have resistance to all known drugs, triggering epidemics of bacterial diseases without a known treatment. Hence, the development of simple and fast methods for determination of residues of multiclass veterinary drugs is required. Liquid chromatography coupled to mass spectrometry ensures great selectivity and detectability for the analysis and become the most used technique for this purpose. However, there are still many bottlenecks on sample preparation due to the different chemical properties of the applied veterinary drugs and the sample composition. Different sample preparation approaches were applied for the extraction of amphenicols, nitroimidazoles, macrolides, quinolones, sulfonamides, benzamides and macrocyclic lactones in salmon muscle. The analysis by UHPLC-MS/MS was applied by using a BEH C18 (50 x 2.1 mm, 1.7 μm) column and mobile phase consisting of water (A) and methanol (B) both with formic acid (0.1%, v/v) and ammonium formate (5 mmol L^{-1}). For the evaluation, spiked samples were extracted and compared to a matrix-matched calibration curve prepared between 1 and 20 μg L^{-1}. Recovery results varied between 15 and 114% with RSD varying between 0 and 38%. The method developed was adequate and the sensitivity achieved meets the MRLs established by regulation for monitoring programs.

P-90   **Quantitative Analysis of Glyphosate in Regular and Infant Grain-Based Foods via LC-MS/MS Using an Anion Exchange HPLC Column**

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While glyphosate remains one of the most commonly used pesticides in the world, it recently came into the focus of the analytical community due to renewed interest from regulatory agencies. This work describes a method for direct analysis of glyphosate in grains after extraction based on the QuPPE methodology using 50:50 methanol:water as an extraction solvent. Cleanup of samples not containing particulates was done using solid phase extraction with HLB. Samples that were cloudy, including wheat flour extracts, required ultrafiltration through membranes with a 3kDa molecular weight cut off (MWCO). The LC-MS method for glyphosate, (aminomethyl)phosphonic acid (AMPA) and glufosinate employed an aminopropyl-bonded polymer-based HPLC column used with carbonate buffer as the mobile phase. The polymer-based column exhibited excellent stability at pH 9, and the carbonate mobile phase was fully compatible with MS detection. The method was validated using spiked samples of organic wheat flour and organic oatmeal in which glyphosate was not detected. The method was applied to both regular and infant cereal and flour samples. Glyphosate was detected in non-organic infant oat cereal at 1 ppm, in infant mixed cereal at 0.25 ppm, in instant oatmeal at 1.2 ppm and in bleached wheat flour at 0.8 ppm. These values fall within the regulatory limits for glyphosate in cereal grains in the USA.

P-91   **Development of Automated Sample Preparation Method for Pesticide Analysis in Baby Foods Using Solid Phase Microextraction with Analysis by GC-MS**

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Solid phase microextraction (SPME) is a technique that can be used for the analysis of a wide variety of analytes in many different sample matrices. For analytes of low volatility, such as pesticides, an immersion of the SPME fiber into a sample is required for extraction. High background samples, such as many foods, pose a challenge to this technique due to the presence of fats, sugars, pigments and other macromolecules. These can stick to the fiber and reduce its usable life and/or be transferred to the GC, where they may interfere with chromatographic analysis. By resolving these challenges related to the immersion technique, the simplicity of automation for SPME methods can be utilized to streamline, simplify, and reduce the cost of sample preparation. In this work, ruggedness of immersion SPME was improved through use of an overcoated SPME fiber in combination with a post-extraction wash step. Optimization of parameters in the SPME method such as sample pH adjustment, salt addition, sample dilution, and extraction temperature were undertaken to achieve accurate and reproducible analyses of pesticides in baby food at low ppb levels.   Results will be presented showing the final SPME method applied to samples spiked with pesticides included on the list described as part of EU directive 2006/125/EC.

P-92   **Analysis of Pesticide Residues in Green Tea Using QuEChERS Extraction and Cleanup with a New Dual Layer SPE Cartridge**

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In the analysis of tea for pesticide residues, QuEChERS is commonly used for extraction. The resulting extract often has high background, requiring some type of cleanup prior to chromatographic analysis. Approaches for cleanup include QuEChERS with a mixture containing graphitized carbon black (GCB), or SPE with dual layer cartridges containing GCB. For both cleanups, GCB removes green pigments, but will also retain any pesticides with planar structures. In the case of SPE, toluene can be added to the elution solvent to increase recoveries. While having toluene present in the final extract is compatible with injection into a GC system, a solvent exchange must be done prior to HPLC analysis.
In this application, cleanup of green tea extracts was achieved using a small volume, dual layer SPE cartridge containing a mixture of a specially engineered carbon, PSA and C18 in the top layer, and Z-Sep in the bottom layer. This cartridge was designed to provide cleanup and pigment removal, with weaker retention of planar pesticides than conventional GCBs. The cartridge used less solvent than typical SPE cleanup protocols, and did not require the use of toluene in the elution solvent. Spiking studies of green tea were conducted at 5 and 50 ng/g, with the resulting extracts cleaned by QuEChERS using PSA/C18/GCB, and SPE using the new cartridge. Analysis for targeted pesticides was then conducted by GC/MS/

MS and LC/MS/MS on the same set of extracts. Performance of the cleanups was compared with regards to background reduction and pesticide recoveries.

P-93   Reducing Matrix Effects in LC-MS/MS Analysis using a Fully Automated Column Switching System

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Analyzing chemical residues in complex sample matrices using LC-MS(/MS) the impact of chromatography is often underestimated. However, co-extracted matrix components from previous LC runs can also collect on the separation column. Even after long flushes with organic solvent, lingering matrix may lead to unexpected irreproducible signal suppression or enhancement during subsequent chromatographic runs. As a result, quantification could become almost impossible, particularly in the case of using Electrospray Ionization (ESI). Not only does the presented column switching system contribute to an accurate quantitation removing lingering components entirely, but also the fully automation increases the sample throughput enormously. Furthermore, a successfully applied use of paired identical analytical columns allows chromatography on the first column while simultaneously rinsing a second separation column after a run had been finished. As an additional outcome a consequently implemented back-flushing extends the LC column lifetime significantly. Moreover, the independent selection of up to 9 LC columns (plus an additional bypass) accompanied by a free choice of different mobile phases enables a fully automated workflow. Therefore, nearly unlimited use of different LC methods is feasible. Thus, manual operation exchanging columns or eluents is no longer needed as well as unattended automated operation overnight and on weekends is achievable. Conclusively, the described column switching system fulfills the requirements of analytical chemists to analyze a large amount of samples with a high performance, efficiency and simplicity. The universal use for food, environmental, proteomics and metabolomics analysis completes the field of applications.

P-94   MRMHR: A Novel Scan Mode for Trace Level Pesticide Analysis of Cannabis Plant Extracts using High Resolution QTOF Mass Spectrometry

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State regulations for pesticides in cannabis products have a zero tolerance approach whereby any pesticide found that is not approved causes the material to be out of compliance which may result in removal of the product from distribution. Compliance with regulations for pesticides is most often performed using LC-MS/MS with a triple quadrupole mass spectrometer but the complex matrix of these samples often can make compound confirmation via ion ratios difficult. We have been investigating the use of a QTOF mass spectrometer using the MRMHR scan mode on a SCIEX X500R QTOF System to provide better confirmative data. With this scan mode it is possible to acquire MRM transitions at optimized collision energies which results in better sensitivity for quantitative applications. We are investigation two different aspects of this scan mode. One is to acquire data over a narrow range of masses which produces data similar to traditional QQQ data. The second is to set up two transitions at optimum collision energies for the fragments but one of the transitions acquires a full scan MS/MS spectrum that can be used simultaneously for quantitation and for compound identification. The results of these studies will be presented. The focus of the presentation will be to show quantitation data for the Oregon list of pesticides and to demonstrate the effectiveness of the MRMHR data for compound identification and target confirmation.

P-95   The Advantages of Using MS/MS ALL with SWATH® Acquisition for Confident Compound Identification in Non Target Screening (NTS) Analyses

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The increased performance of high resolution mass spectrometry instrumentation has allowed chemists to have an unprecedented ability to identify completely unknown compounds in complex matrices. In earlier instrumentation, this has been accomplished with a data independent acquisition. In these types of acquisitions, all of the ions from a wide mass range, 100-1000 for example, are allowed into the fragmentation chamber and the resulting fragments are analyzed. In an effort to generate MS/MS spectra that are library searchable, different workers have developed schemes for de-convoluting the resulting spectra based on differences in retention times. Unfortunately, in the case where compounds completely co-elute it is impossible to generate clean spectra. SCIEX has developed the MS/MS ALL with SWATH® acquisition to provide better spectra from data independent acquisitions. In this presentation SWATH® will be described and the advantage of such an approach will be shown. Data from actual matrices will be presented along with library searching results to demonstrate the improved quality of mass spectra using MS/MS ALL with SWATH® acquisition.

P-96   Application of a High Resolution Product Ion Library to Screen for PFASs

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Perfluoroalkyl substances (PFASs) encompass a range of fully fluorinated alkyl compounds and are prevalent in Aqueous Film Fire Foam (AFFF). In addition, PFASs are ubiquitous as they are used in many household goods and have been found in various environmental and biological samples. Here, we demonstrate the use of QTOF technology to exploit the power of high resolution mass spectrometry and the use of product ion spectra to identify novel compounds in AFFF contaminated samples. The use of library searching to identify unique and unexpected compounds in these samples will be presented.

P-97   Determination of Microcystins In Aqueous Samples By Liquid Chromatography-Tandem Mass Spectrometry

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Microsystins (MCs) are toxins produced by freshwater cyanobacteria or blue-green algae. Because of the health risks associated with MCs, it has been added to the Fourth Unregulated Contaminant Monitoring Rule which will be promulgated in 2018 by the U.S. Environmental Protection Agency (US EPA). Here, we have developed a highly sensitive method to analyze trace amounts of MCs in water. The method presented includes Solid Phase Extraction coupled to liquid chromatography-tandem mass spectrometry for the determination of part per trillion concentrations of the MCs required by USEPA Method 544.

P-98   SFC-HRMS a novel analytical technique for cannabinoids analysis in hemp oil

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Cannabinoids represent a group of biologically active chemicals naturally occurring in *Cannabis* plants. With regards to a global interest in these compounds (including medicine), the accurate analysis of their levels has become of high concern not just in green part of plant but also in hemp oil. All plants in the *Cannabis* genus can produce the oil, but usually only industrial hemp (with a minimum of the psychoactive substances) is used to make hemp oil. Hemp oil is typically almost free of THC, but this claim is necessary to verify by appropriate analytical tool.
Currently, liquid chromatography coupled with mass spectrometry (LC-MS) is a preferred technique used for cannabinoids analysis. In case of oil, the presence of triacylglycerols (TAGs), in final extract, represents a problem due to their strong retention in the LC-MS system when the reverse phase is used. Within this study a novel strategy based on supercritical fluid chromatography coupled with high resolution mass spectrometry (SFC-HRMS) was used. This analytical strategy solve the problem with retention of TAGs in analytical system, because in case of SFC-HRMS carbon dioxide (non-polar mobile phase) is used and TAGs are easily eluted. As a result optimized and validated method for the determination of nine cannabinoids (CBD, Δ9-THC, CBG, CBDV, CBDA, THCA, CBGA, CBN, Δ8-THC) in hemp oil was developed. Developed method

allows the measurement of all analytes in the same ionization mode and the satisfactory validation parameters, recovery (71-110%) and repeatability expressed as RSD (2-12%), were achieved.

P-99   Simultaneous determination of perfluoroalkyl substances (PFASs) and organophosphorus flame retardants (OPFRs) in dust

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The presented study was focused on a development of a new analytical approach for simultaneous determination of 16 organophosphorus flame retardants (OPFRs) and 22 perfluoroalkyl substances (PFASs) in indoor dust. In the first step, the potential of ultra-high performance liquid (UHPLC) and gas chromatography (GC) coupled with tandem mass spectrometry (MS/MS) for the determination of OPFRs was assessed. Using UHPLC–MS/MS, significantly lower limits of quantification (0.05–0.5 pg in injection) compared to GC–MS/MS (0.8–80 pg in injection) for OPFRs were achieved, thus this technique was applied for determination of both contaminants'´ groups. The biggest challenge in the analysis of these emerging contaminants is their ubiquitous presence in the laboratory environment even in chromatographic systems, which could cause an overestimation of their results. For this reason, a great attention was focused on simplification of the analytical procedure to minimize the risk of a sample contamination. The tested methods were ultrasonication employing various solvents (methanol, hexane and ethyl acetate) and QuEChERS approach. Comparing the extraction efficiency of mentioned solvents, as well as the matrix effects, ultra-sonication with methanol was selected for dust analysis, with recoveries of OPFRs and PFASs in the range of 90–110% and repeatability less than 15%. Moreover, this simple approach without no discriminative purification step allows further non-target screening for the comprehensive assessment of human exposure to a wide spectrum of chemicals associated with indoor dust.
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P-100   Streamlined Method for EPA Method 1694: Pharmaceuticals and Personal Care Products in Water

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EPA Method 1694 is a non-regulatory screening method for the analysis of pharmaceuticals and personal care products (PPCPs) in environmental samples, including water. The method uses SPE and LC-MS/MS to analyze 73 PPCPs in drinking water, surface water and treated wastewater. The PPCPs include common prescription drugs, over-the-counter medicines, dietary supplements and consumer products. The method divides the PPCPs into four groups based on their physicochemical properties. Water samples are extracted via two SPE procedures (pH 2 and pH 10) and LC-MS/MS analysis is carried out with four separate methods that use different HPLC columns, mobile phases, gradients and ionization modes (ESI+ and ESI-). Fortunately, modifications to EPA 1694 are allowed if they provide performance equal to or better than that specified in the official method. This poster outlines a streamlined analytical method for EPA 1694. The SPE procedure was optimized to achieve acceptable recoveries of the PCPPs using a single extraction step rather than the multiple extraction procedures outlined in the original EPA method. Water samples are extracted using a highly cross-linked polymeric SPE cartridge (Enviro-Clean® HL DVB) without any pH adjustment. LC-MS/MS analysis uses a single HPLC column (Selectra® DA) and two methods (ESI+ and ESI-) rather than the two HPLC columns and four methods outlined in EPA 1694. The recovery and RSD values obtained were found to be within the method requirements for the vast majority of PPCPs. Overall, this streamlined approach significantly reduces the analysis time while still achieving comparable results to the original EPA method.

P-101   Determination of β-agonists, including ractopamine, in animal tissues and urine by liquid chromatography-tandem quadrupole mass spectrometry

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Although the administration of β-agonists as growth-promoting agents in food-producing animals is banned in many countries due to concerns over human health, ractopamine is authorized for some animal production in a limited number of countries. As these countries have significant export trade of meat to countries where β-agonists are banned, split production systems with associated certification programs have been put in place. Countries where use

is approved (*e.g.* USA and Canada) have maximum residue limits (MRLs) or tolerances for ractopamine, whereas those that have banned meat products containing β agonists have to take an alternative approach. As no Minimum Required Performance Limits (MRPLs) were set for β-agonists, the EU relies on Recommended Concentrations to indicate required performance of analytical methods used for those substances. In contrast, Russia, also with a zero tolerance policy, established an action level for ractopamine in imported consignments of meat one hundred times lower than the Codex MRL.

Monitoring compliance with these limits requires the use of highly sensitive and selective analytical methodology based upon liquid chromatography-tandem mass spectrometry (LC-MS/MS). Examples of performance are given for the analysis of 16 β-agonists in various animal target tissues (*e.g.* liver), edible products (*e.g.* muscle) and urine collected from animals on the farm. As the frequency of detection of residues is low, samples are screened using an adequate level of quality control including the use of stable isotope analogues as surrogates to monitor sample-to-sample variability in matrix effects.   Any suspect positives are re-analyzed with a suitable confirmatory method.

P-102   Rapid Pass-Through Cleanup of Bovine Liver Samples Prior to UPLC-MS/MS Multiresidue Veterinary Drugs Analysis

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Tissue samples, such as bovine muscle and liver, are typically extracted with an acetonitrile based solvent for LC-MS determination of veterinary drug residues. Among the most significant co-extracted substances are fats and polar lipids, particularly phospholipids (lecithin). Bovine liver typically contains about 45 mg of fat and about 25 mg of phospholipids per gram of tissue. Fats can be effectively removed from the acetonitrile based tissue extracts by liquid extraction with hexane or with SPE with octadecyl silica (C18).   However, these defatting procedures are not effective for removal of phospholipids. Excessive amounts of phospholipids can shorten LC column life, contribute to ion-suppression, and contaminate the mass-spectrometer. In this study a novel reversed-phase sorbent is used for highly effective removal of both phospholipids and fats from bovine liver extracts prior to LC-MS/MS analysis. Greater than 95 % of phospholipids and greater than 85 % of fats were effectively removed from the tissue extracts after the simple pass-through SPE procedure. Recoveries of 45 compounds with published MRLs in beef liver averaged 83% with only a few compounds under 60%.

P-103   A New Strategy for the Determination of Captan and Folpet in Food Matrices

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Screening food samples for contaminants, such as pesticides, requires the use of both GC-MS and LC-MS techniques. In order to cover a full suite of regulated compounds, several LC and GC methods are usually required that separately incorporate large suites of compounds, single residues and 'troublesome' compounds. Phthalimide fungicides, including captan and folpet, are compounds that are considered GC amenable pesticides, but are troublesome to analyze via standard GC analysis. In matrices containing chlorophyll, captan and folpet rapidly degrade in the injector port, making repeatable and robust analysis of these compounds very difficult. In this analysis, two LC ionization techniques (Electrospray and UniSpray) were assessed as to their ability to incorporate these problematic GC compounds into an LC method. Initial results show that captan and folpet can be analyzed in food matrix samples using an LC method using both ionization techniques. Method optimization was performed independently on each ionization technique prior to sample analysis. Linearity was assessed with the matrix samples having R^2 values of > 0.99 for both compounds. The methods were also shown to be reproducible with matrix RSDs below 15%. The data presented demonstrates that the notoriously troublesome GC compounds captan and folpet can be analyzed using an LC-MS/MS via ESI or UniSpray ionization techniques.

P-104   Multiresidue Pesticide Analysis in Fruit and Vegetable Commodities Using Both UPLC and APGC on a Single Mass Spectrometer Platform

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Consumer concerns and federal regulations make pesticide residue analysis an important component of ensuring food safety. It is desirable to rapidly and reliably screen samples for a large number of pesticides in as few methods as possible. Comprehensive pesticide screening is typically performed using both LC-MS and GC-MS techniques on dedicated MS platforms. For this analysis, both UPLC-MS/MS and APGC-MS/MS pesticide residue analysis was performed on a single mass spectrometer, with a changeover time from LC-MS/MS to GC-MS/MS of less than 30 minutes. The same sample extracts prepared from various fruit and vegetable commodities were run on both chromatography systems that were coupled to the same tandem quadrupole mass spectrometer. Each method targeted a list of approximately 200 compounds each, monitoring for at least two MRM transitions for each compound. Standards for data quality were taken from the SANTE Guidelines (11945/2015). In the four matrices analyzed, >96% of compounds were detected at 10 µg/kg, with a majority of compounds detectable below 1 µg/kg on both UPLC and APGC. The coefficient of determination on matrix extracted calibration curves were generally > 0.995 and ion ratios fell within 30% of the reference value. The RSD was < 10% for upwards of 90% of the compounds detected at 10 µg/kg. The data presented demonstrates the ability to increase compound coverage on a single mass spectrometer with the flexibility and reliability of performing UPLC-MS/MS and APGC-MS/MS for routine multiresidue pesticide analysis.

P-105   Evaluation of Novel Dispersive SPE and Pass-Through SPE Options for Cleanup of High Chlorophyll Samples after QuEChERS extraction

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In recent years, food safety laboratories have adopted new and simplified sample preparation methods, such as QuEChERS, to reduce analysis time and to increase throughput. When spinach or other leafy vegetables are subjected to QuEChERS extraction, significant amounts of chlorophyll and other pigments are co-extracted along with the target pesticides. The presence of these co-extracted substances can lead to chromatographic interference, contamination of GC or LC systems, and contamination of the mass spectrometer. To avoid these complications, a cleanup step is recommended prior to the instrumental analysis. For spinach, this is often performed using dispersive SPE (dSPE) with graphitized carbon black (GCB) or other sorbents designed to remove chlorophyll. In this study, chlorophyll and other natural pigments in a raw spinach sample, such as lutein and carotene, were measured using UHPLC with photodiode array detection (PDA). Then various types of dispersive and pass-through SPE cleanup options were evaluated for pigment removal (monitored using PDA) and pesticide recovery (monitored using GC-MS/MS and LC-MS/MS). Particular attention was given to recoveries of planar pesticides commonly used on spinach.

P-106   Direct analysis of glyphosate and similar polar pesticides in various food matrices

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A panel of representative polar pesticides has been targeted in a single liquid chromatographic tandem quadrupole mass spectrometry method, namely aminomethylphosphonic acid (AMPA), chlorate, ethephon, fosetyl aluminium, glufosinate, glyphosate, maleic hydrazide and phosphonic acid. Extracts of various foodstuffs were prepared in accordance with the Quick Polar Pesticides (QuPPe) extraction method. Chromatographic separation was achieved on mixed mode / hydrophilic interaction liquid chromatography (HILIC), applying a mobile phase gradient. Targeted multi-reaction monitoring methods were used, with at least two transitions per compound to quantify and confirm analyte detection. Chromatographic performance was evaluated in accordance with SANTE guideline document 11954/2015 columns, certain limitations were determined for the challenging analysis of these highly polar pesticides. This evaluation has for a LC-MS/MS method for the direct analysis of anionic pesticides. Retention of all analytes was greater than two times the void volume, while the retention time tolerance for each analyte was within ±0.1 minute of the corresponding matrix matched standard.
Overall method performance, in the absence of isotopically labeled internal standard, was evaluated by assessing linearity, accuracy and sensitivity.

P-107   A Novel Data-Independent Acquisition Strategy for Non Targeted, Accurate Mass Contaminant Screening

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Companies and environmental regulatory authorities continue to investigate High Resolution Mass Spectrometry (HRMS), non-targeted, screening techniques to expand the scope of their screening methods. Improvements in mass spectrometer sensitivity and highly selective acquisition techniques, alongside advancements in the informatics used to process and review data, are facilitating the task.

Several fruit and vegetable samples, previously characterized by a collaborator, were screened against a pesticide library using several Data-Independent-Acquisitions (DIA). DIA strategies for complex mixture analysis, particularly within a contaminant screening environment offer significant efficiency advantages in that they enable a generic, non-biased strategy for data acquisition. The work will discuss the use of a novel DIA method – SONAR, in which precursor and product ion data are acquired with a sliding quadrupole window - for use in accurate mass screening applications and its use alongside more traditional DIA strategies such as full scan low and high collision energy acquisition (MS^E), and its ion-mobility enhanced variant (HDMS^E).

A comparison of the outcomes from MS^E, HDMS^E and SONAR screening sets will be presented.

All techniques were able to detect contaminants present. However, the comparison of each technique with increasing matrix complexity shows that the ability of non-selective full scan experiments (MS^E) to generate clean product ion spectra is reduced when compared to HDMS^E and SONAR datasets.

P-108   The analysis of natural and synthetic estrogens at low ppq levels in surface water and final effluent water by liquid chromatography-tandem quadrupole mass spectrometry

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Estrogens are routinely used either as contraceptive medicines or in hormone replacement therapy and can enter aquatic environments via the discharge of final effluent waters. Estrogens are believed to have a negative effect on aquatic environments by disrupting the hormonal systems of fish. In EU directive 2013/39/EU fifteen additional priority substances were added to the Water Framework Directive (2000/60/EC) and 17α-ethinylestradiol and 17β-estradiol added to a watch list in order to gather further data on the presence of these compounds in aquatic environments. In this presentation we highlight a method for the analysis of synthetic estrogens in surface and final effluent waters at low ppq levels using LC-MS/MS.

Spiked surface water and final effluent samples were extracted and concentrated using solid phase extraction (SPE). After evaporation and reconstitution in water, the samples were then analyzed by LC-MS/MS using a large volume injection (100μl). The method's performance was evaluated by assessing linearity, repeatability, sensitivity and recovery. Satisfactory quantitative performance was achieved for all compounds in both surface water and final effluent over appropriate concentration ranges (linear, R^2 > 0.997, residuals < 15%). Recoveries from surface water were ≥70% and precision good (RSDs <6%). The method showed high sensitivity, achieving the required European (2015/495/EU) LLOQ (PtP s/n=10) levels for each compound in matrix. Low level concentrations of 17α-ethinylestradiol were detected in the final effluent matrix and a standard addition method was used to calculate the concentrations.