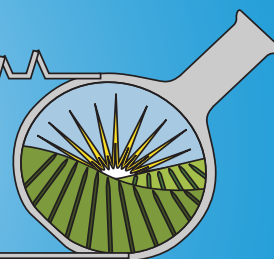




56th Annual

NACRW



NORTH AMERICAN CHEMICAL RESIDUE WORKSHOP

"Bringing Scientists Together to Develop and Validate Better Methodologies"



Powerful Platforms for Trace-Level Analysis of Pesticides, Mycotoxins, and Other Contaminants in Food and Environmental Samples

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LCMS-8060 Triple Quadrupole Mass Spectrometer

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GCMS-TQ8050 NX Triple Quadrupole Mass Spectrometer

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FUTURE MEETING DATES

2020 July 26-29
Marriott Harbor Beach Resort
Fort Lauderdale, Florida

2021 July 25-28
Marriott Harbor Beach Resort
Fort Lauderdale, Florida

2022 July 24-27
Marriott Harbor Beach Resort
Fort Lauderdale, Florida



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Dear Attendees, Exhibitors, Sponsors and Guests;

Welcome everyone to the 56th, 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!

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. We invite you to attend our Monday evening on-site social event! We've planned a Caribbean-themed meal, 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 NACRW Excellence Award, the award topic is Excellence in Detection Techniques. We are pleased to announce the recipient of the 2019 NACRW Excellence Award is Michael Story, who with Robert Finnigan, were early pioneers in the development of quadrupole mass spectrometers for commercial use. Their instruments became the gold standard technology for analytical labs around the world.

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 and veterinary drugs. In addition, special topic sessions on natural products, cannabis, emerging contaminants, single and multi-residue methods, and validation/verification will be featured. We also have the very informative and popular Updates from the Federal and State Regulatory Laboratories. Based on your feedback from last year's survey, the program committee decided to change the topic of the open forum session from Mass Spectrometry to Pesticide Residues. Remember this open forum on Tuesday afternoon is driven by you, so please be sure to submit your questions to the moderators or to the Q&A box located by registration table.

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. 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 2019 NACRW Organizing Committee, Program Committee, especially Brittany Holmes, Ping Wan, 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,

Ken Kise, 2019 Organizing Committee President
Ping Wan and Brittany Holmes, 2019 Program Committee Co-Chairs
2019 Organizing Committee and Program Committee members



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 so many hours that contributed to the Advancement of NACRW.

Past Recipients

2011 George and Wilma Fong-Founders

2012 Gail Parker

2013 Pat Beckett

2014 Sherry Garris

2015 Jack Cochran

2016 Amy Brown

2017 Jo Marie Cook

2018 Julie Kowalski

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Sherry Garris, Chair
Jack Cochran
Jo Marie Cook, Treasurer
Susan Eigen

Danny LeCompte
Gail Parker
Sherri Turnipseed
Jon Wong
Teri Besse, Executive Director



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Co-Chairs:

Brittany Holmes, WA State Dept. of Agriculture
Ping Wan, Office of Indiana State Chemist

Co-Chairs Elect:

Katie Carlos, US FDA CFSAN
Mike Filigenzi, CAHFS, U.C. Davis

Immediate Past Co-Chairs:

Mark Engel, FDACS
Robert Trengove, Murdoch University

Program Committee Members

Neely Belai, US FDA
Katie Carlos, US FDA CFSAN
Martin Dušek, Research Institute of Brewing and Malting
Mark Engel, FDACS
Ken Kise, Iowa Dept. of Agriculture
Julie Kowalski, Trace Analytics
Jessica Krank, Colorado Department of Agriculture
Susie Genualdi, US FDA
Yoko Johnson, MN Dept Of Agriculture Lab Services Div
Katerina Mastovska, Eurofins
Sara McGrath, US FDA
Lynda Podhorniak, EPA/Office of Pesticide Programs
Paul Reibach, Smithers Viscient
Sherri Turnipseed, US FDA/ORR/ADRC
Eric Verdon, ANSES
Jon Wong, US FDA

Poster Committee

Co-Chairs

André de Kok, Wageningen Food Safety Research
Brittany Holmes, WA State Dept. of Agriculture

Poster Committee Members

Kelly Dorweiler, General Mills, Inc.
Brian Eitzer, The Conn. Agr. Exp. Station
Mark Engel, FDACS
Sherri Turnipseed, US FDA/ORR/ADRC

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Ken Kise, Iowa Dept. of Agriculture

Secretary:

Alexandria Pavkovich Bush, Restek Corporation

President-Elect:

Paul Reibach, Smithers Viscient

Immediate Past President:

Alex Krynitsky, Symbiotic Research LLC

Organizing Committee Members

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Brad Barrett, LECO Corporation
Emily Britton, Waters Corporation
Rodney Bennett
Oscar Cabrices, SCIEX
Poonam Chandra, CA Department of Food & Agriculture
Jack Cochran, VuvAnalytics
Jo Marie Cook
Brian Eitzer, The Conn. Agr. Exp. Station
Sherry Garris
Simon Hird, Waters Corporation
Robert Hunsberger, NV Department of Agriculture
Nathan Johnson, Campbell Soup Company
Joseph Kolb, Merieux Nutrisciences
Joe Konschnik, Restek Corporation
Scott Krepich, Phenomenex
Steve Lehotay, USDA ARS
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Cassidy Miller, Agilent Technologies
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Adam Ross, LGC
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Jody Searfoss, UCT
Dimple Shah, Waters Corporation
Tameka Taylor, Environmental Protection Agency
Ryan Undeen, Merieux Nutrisciences
Jona Verreth, Montana Dept. of Agriculture
Jian Wang, Canadian Food Inspection Agency
Philip Wylie, Agilent Technologies
Paul Yang, Ontario Ministry of the Environment

Sub-Committee Groups

Communications Committee

Chair: Alexandria Pavkovich Bush
Emily Britton
Rodney Bennett
Julie Kowalski
Jessica Krank
Jody Seafoss
Dimple Shah

Social Event Committee

Chair: Oscar Cabrices
Stacy Aylesworth
Nathan Johnson
Joe Konschnik
Scott Krepich
Sareeta Nerkar
Dimple Shah

Excellence Award Committee

Chair: Ken Kise
Brad Barrett
Jack Cochran
Steven Lehotay
Sareeta Nerkar
Alexandria Pavkovich Bush
Paul Reibach
Yelena Sapozhnikova

Student Scholarships Committee

Chair: Katie Carlos
Emily Britton
Poonam Chandra
Jack Cochran
Scott Krepich
Alexandria Pavkovich Bush
Ryan Undeen

Short Course Committee

Chair: Joe Konschnik
Marc Engel
Simon Hird
Scott Krepich
Dimple Shah
Eric Verdon

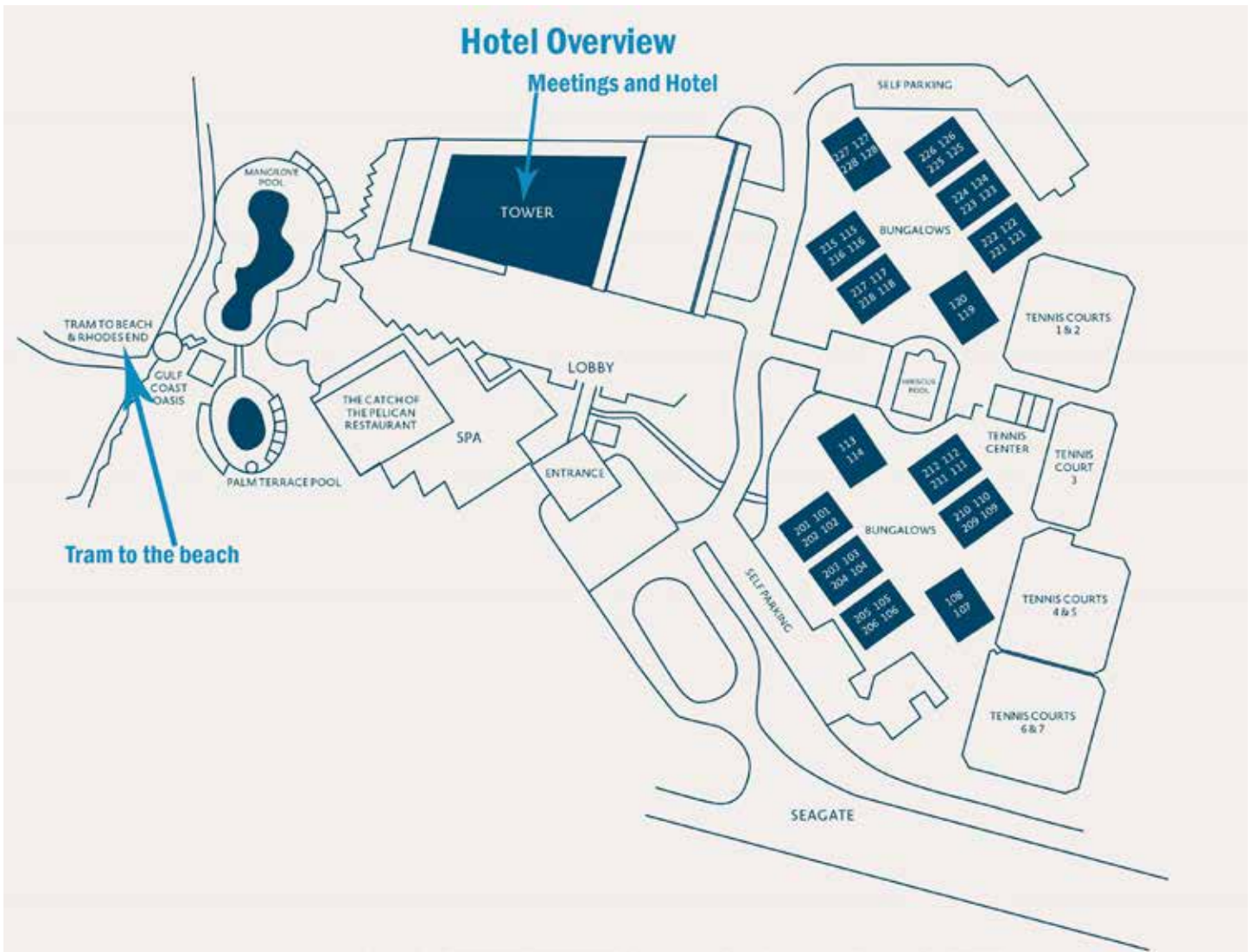
Thank You

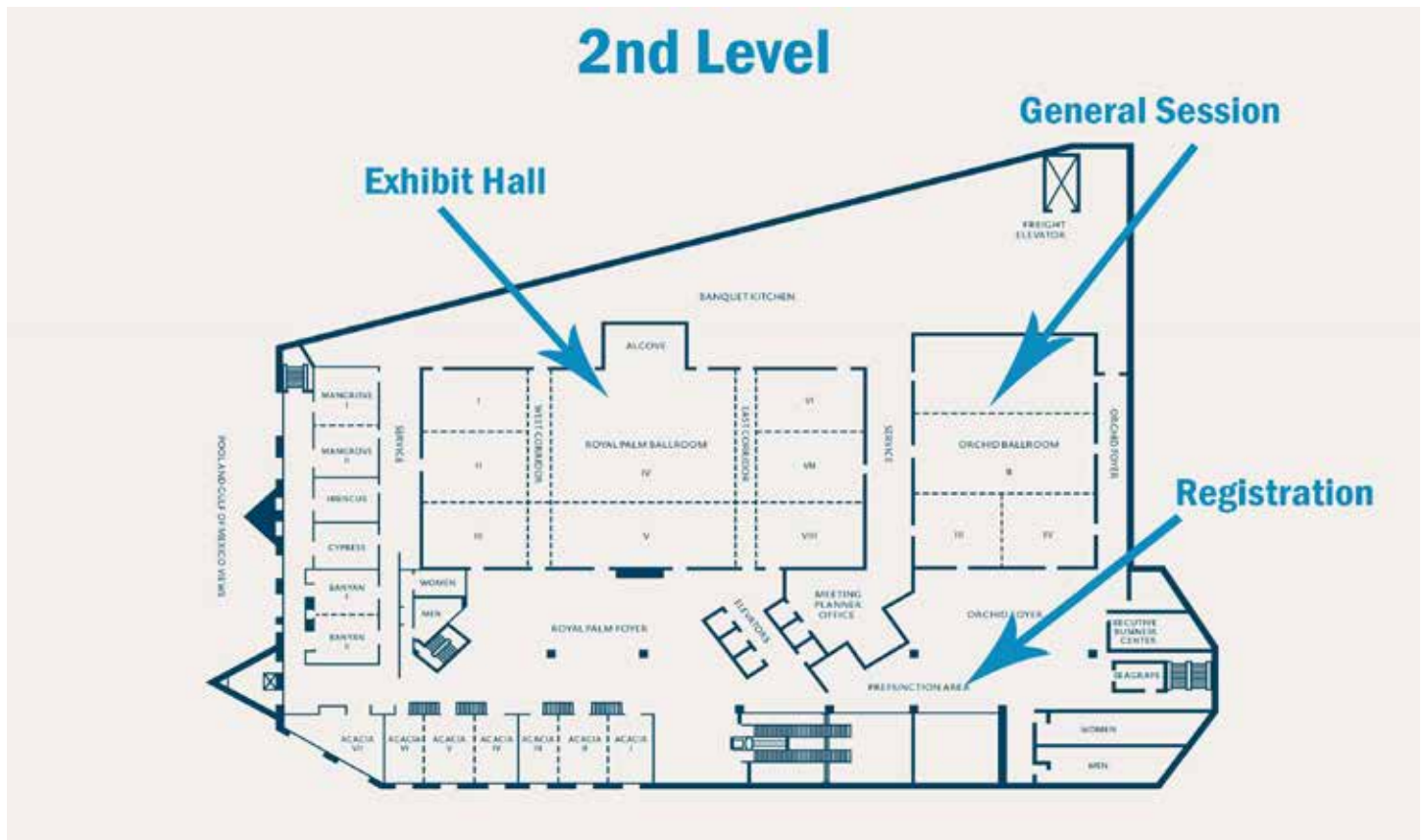
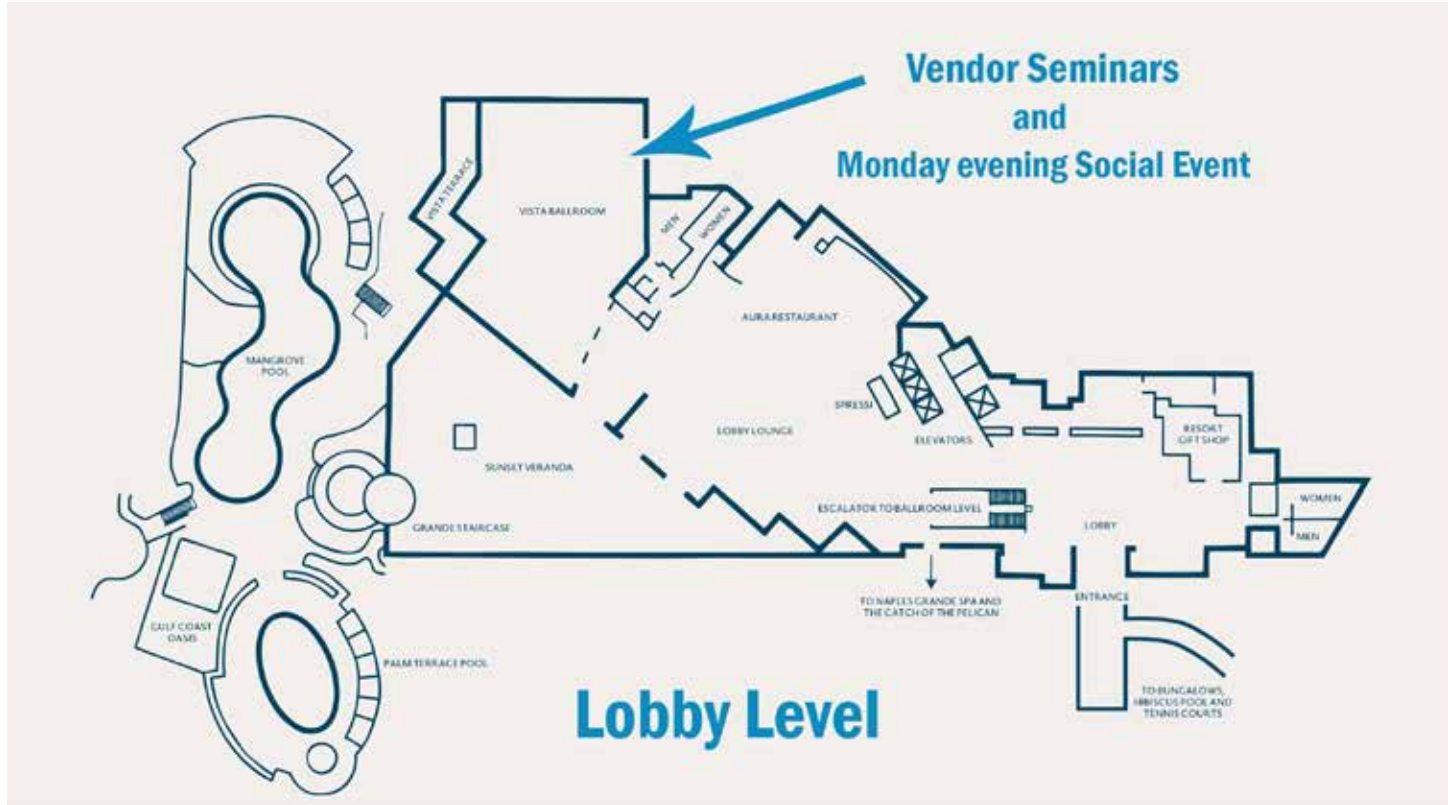
to all of our amazing volunteers!



NAPLES GRANDE

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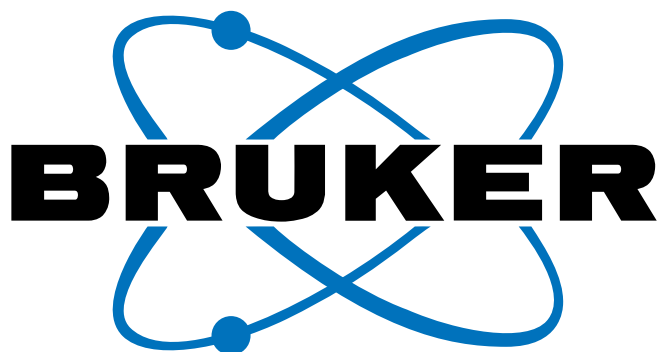


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MEETING AT A GLANCE

Sunday, July 21, 2019

noon-6:00 pm	Registration	Orchid Foyer
1:00-5:00 pm	Exhibitor Setup	Royal Palm Ballroom
1:00 - 2:30 pm	NACRW Reference Materials Working Group	Orchid 1-2
2:45 - 4:15 pm	NACRW Veterinary Drugs Working Group	Orchid 1-2
4:30 - 6:00 pm	PERFORM Working Group	Orchid 1-2
3:00-6:00 pm	Poster Board Set Up	Royal Palm Ballroom
6:15-7:15 pm	Shimadzu Scientific Instruments, Inc. Vendor Seminar	Vista Ballroom, 1st level
7:30-9:30 pm	Welcome Reception	Royal Palm Ballroom

Monday, July 22, 2019

7:30 am-5:00 pm	Registration	Orchid Foyer
7:00-10:00 am	Poster Board Set Up	Royal Palm Ballroom
7:15-7:45 am	Moderator and Volunteer Training	Orchid Ballroom
7:15-8:15 am	Waters Corporation Vendor Seminar	Vista Ballroom, 1 st level
7:30-8:15 am	Early Morning Coffee	Orchid Foyer
8:30-8:40 am	Opening Remarks Sherry Garris, Chair, NACRW Board of Directors	Orchid Ballroom
8:40-8:45 am	NACRW Excellence Award Presentation and Keynote Address Introduction and Presentation of NACRW Excellence Award Ken Kise, 2019 NACRW President	
8:45-9:30 am	Presentation by Excellence Award Winner	
9:30-10:45 am	SESSION 1: Single Residue and Small Scale Multiresidue Methods Co-Chairs: Katherine Carlos and Lynda Podhorniak	Orchid Ballroom
10:45-noon	Exhibition and Poster Opening	Royal Palm Ballroom
11:00-noon	Poster Session A (<i>authors present for odd #s</i>)	Royal Palm Ballroom
noon-1:00 pm	Cash Lunch (Exhibition Hall)	Royal Palm Ballroom
12:15- 1:15 pm	Agilent Vendor Seminar	Vista Ballroom, 1 st level
1:30-3:10 pm	SESSION 2: Method Validation and Verification Co-Chairs: Paul Reibach and Kate Mastovska	Orchid Ballroom
3:10-3:55 pm	BREAK (Exhibition & Posters)	Royal Palm Ballroom
3:55-5:45 pm	SESSION 3: Analysis of Cannabis Co-Chairs: Julie Kowalski and Jessica Krank	Orchid Ballroom
6:30-9:30 pm	SOCIAL EVENT – Naples Grande	Sunset Veranda/Vista Ballroom

Tuesday, July 23, 2019

7:30 am-5:00 pm	Registration	OrchidFoyer
7:30 am-4:00 pm	Exhibition & Posters	Royal Palm Ballroom
7:00-8:15 am	Bruker Vendor Seminar	Vista Ballroom, 1st level
7:30-8:15 am	Early Morning Coffee	Royal Palm Ballroom

MEETING AT A GLANCE

8:45-10:45 am	SESSION 4: Multi-residue Methods for Pesticides and Related Contaminants Co-Chairs: Martin Dusek and Jon Wong	Orchid Ballroom
10:45-noon	Exhibition and Poster Opening	Royal Palm Ballroom
11:00-noon	Poster Session B (<i>authors present for even #s</i>)	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, 1 st level
1:30-3:10 pm	SESSION 5: Novel and Emerging Contaminants Co-Chairs: Susie Genualdi and Neely Belai	Orchid Ballroom
3:10-3:55 pm	BREAK (Exhibition & Posters)	Royal Palm Ballroom
3:55-5:00 pm	SESSION 6: Pesticide Residue Forum Moderators: Walter Hammack, Simon Hird, Brian Eitzer	Orchid Ballroom
5:05-6:00 pm	NACRW Organizing Committee Meeting <i>open to all attendees</i>	Orchid Ballroom
6:00-10:30 pm	Shuttle service to and from Mercato (Blue Martini) <i>one shuttle scheduled to run every 15 minutes</i>	Outside - Hotel Main Entrance

Wednesday, July 24, 2019

7:30 am-noon	Registration	Orchid Foyer
7:30 am-noon	Exhibition & Posters	Royal Palm Ballroom
7:15-8:15 am	SCIEX Vendor Seminar	Vista Ballroom, 1 st level
7:30-8:15 am	Early Morning Coffee	Royal Palm Ballroom
8:30-10:45 am	SESSION 7: State/Federal Lab Updates Co-Chairs: Yoko Johnson and Ken Kise	Orchid Ballroom
10:30-10:45 am	Student Scholarship Presentations	Orchid Ballroom
10:45-noon	BREAK (Exhibition & Posters)	Royal Palm Ballroom
12:00-1:00 pm	Pickering Laboratories Vendor Seminar	Vista Ballroom, 1 st level
1:05-2:45 pm	SESSION 8: Trends in Veterinary Drug Residue Control Co-Chairs: Sherri Turnipseed and Eric Verdon	Orchid Ballroom
2:45-3:15 pm	BREAK	Orchid Foyer
3:15-4:55 pm	SESSION 9: Emerging Topics in Trace Level Analysis Co-Chairs: Marc Engel	Orchid Ballroom
4:55-5:10 pm	Poster Awards and Closing	Orchid Ballroom

Thursday, July 25, 2019

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



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GENERAL INFORMATION

Registration

Check in once at the registration desk at your earliest opportunity

Sunday - noon – 6:00 pm

Monday - 7:30 am – 5:00 pm

Tuesday - 7:30 am – 5:00 pm

Wednesday - 7:30 am – Noon

KEY to Presentation Numbering System

Oral presentations are numbered O-1, O-2, O-3, O-4, etc.

Vendor Seminars are numbered V-1, V-2, V-3, V-4, etc.

Session A posters are ODD numbered P-1, P-3, P-5, etc.

Session B posters are EVEN numbered P-2, P-4, P-6, etc.

Poster Sessions (Exhibit Hall, Royal Palm Ballroom)

Hang posters Sunday afternoon from 3:00 pm to 6:00 pm or Monday morning from 7:00 am to 10:00 am.

Take down posters between 12 noon to 2:00 pm on Wednesday

Posters may be viewed any time Exhibition is open

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

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

Poster Prizes

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

Exhibition

Sunday evening reception with light hors d'oeuvres and an open bar from 7:30 to 8:30 pm and then a cash bar after that until 9:30 pm.

Monday - 11:00 am - 5:00 pm

Tuesday - 7:30 am – 4:00 pm

Wednesday - 7:30 am – noon

Coffee and Breaks

Coffee will be available 7:15 - 8:15 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 24th, 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 2019 NACRW. Please inform **Brittany Holmes, 2019 Program Co-Chair (bholmes@agr.wa.gov)**, by **September 2, 2019**, if you intend to submit an article. Authors will then be invited by JAFAC to submit their manuscripts electronically online through the JAFAC website with a deadline of November 29, 2019.

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 a few years.

Meeting Evaluations

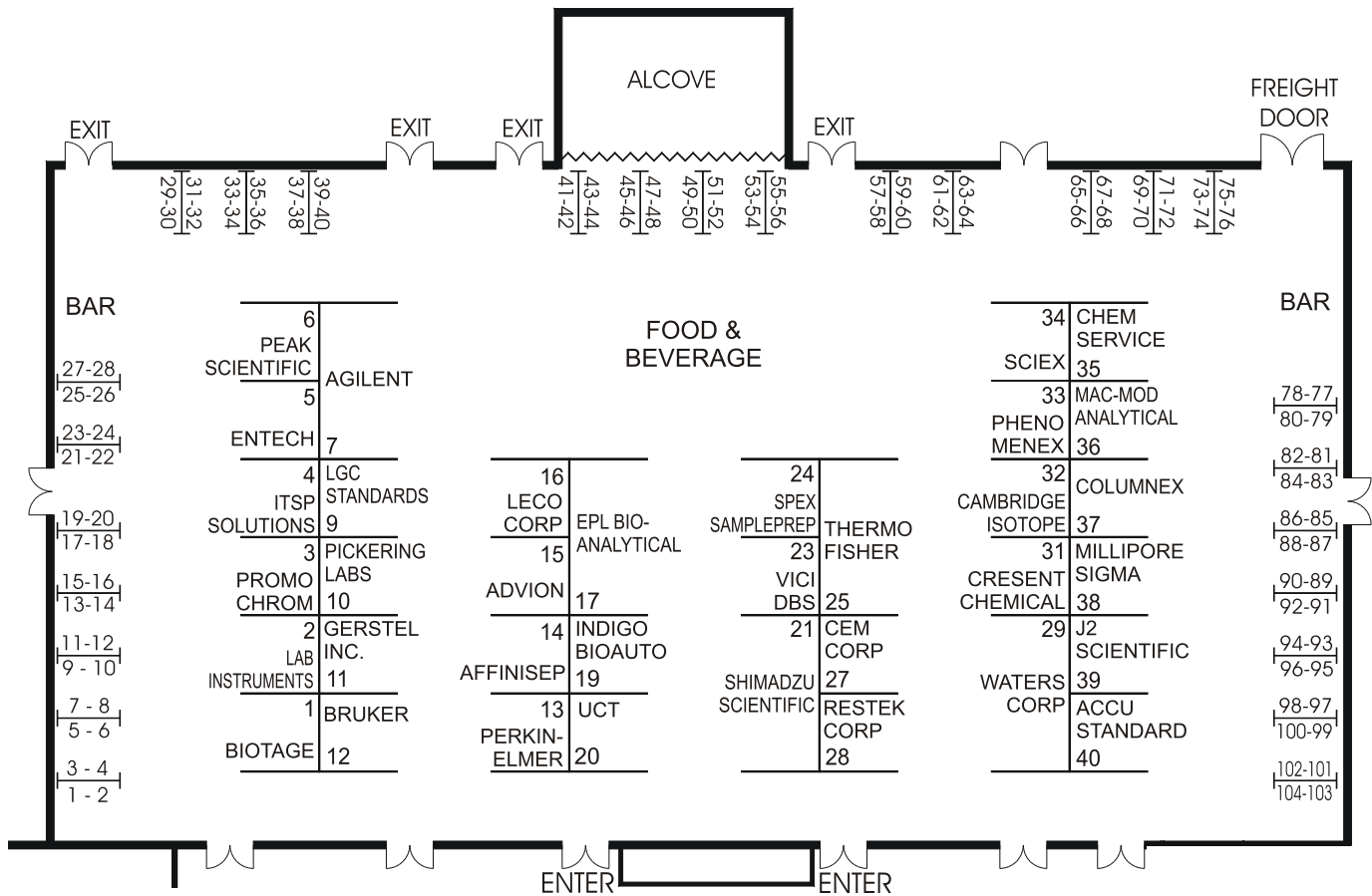
Look for an on-line conference evaluation on the last day of the conference. The evaluation will be emailed to you, so please take a few moments to fill out the online form.

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 2020 NACRW
July 26-July 29, 2020 Fort Lauderdale, Florida - Marriott Harbor Beach Resort

EXHIBITION HALL AND POSTER SESSIONS

Location: Royal Palm Ballroom, 2nd level



EXHIBITORS

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J2 Scientific, LLC
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www.j2scientific.com

Lab Instruments Srl
Booth #2
www.labinstruments.org

LECO Corporation
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LGC Standards
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Mac-MOD Analytical
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MilliporeSigma
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Peak Scientific Instruments LTD
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Perkinelmer
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Booth #29
www.waters.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 21, 2019, 6:15 p.m. to 7:15 p.m. SHIMADZU SCIENTIFIC INSTRUMENTS, INC.
Location: Vista Ballroom

What's in my Hemp? The good, the bad, and the toxic!

*Volker Bornemann, Avazyme, Inc., 2202 Ellis Rd., Suite A, Durham, NC 27703, USA;
volker.bornemann@avazyme.com*

Hemp, also known as *Cannabis sativa*, has been prohibited to be grown in the US for the past 70 years, and was considered for the most part a schedule I controlled substance by the US Drug Enforcement Administration. This has changed at the beginning of this year with the enactment of the 2018 Farm Bill. Now, industrial hemp is “just another crop” in the United States and subject to the usual regulations for crops and crop derived products, like food, feed, cosmetic products, and more. Testing emerges as a critical factor to ensure product safety and product quality, and that the hemp derived products are safe for the environment, humans, pets, and livestock. This includes pesticide uses in hemp, which have to be registered with the US EPA and the individual State regulatory agencies. Testing is also needed to ensure compliance with all applicable laws and regulations. A holistic approach to testing is needed to test for cannabinoid and terpene profiles, adulterants like heavy metals, mycotoxins, pesticide residues, residual solvents, pathogens, nutritional composition and equivalency for ingestible food and feed products, and more.

Avazyme, Inc. is a customized testing solution provider offering field and laboratory testing, product development, and expert consulting services to agriculture and the entire food value chain. Avazyme provides fast, accurate and reliable answers to ensure product safety and high product quality for food, feed, cosmetics, nutraceutical, and pharmaceutical products.

V-2 Monday, July 22, 2019, 7:15 to 8:15 am WATERS CORPORATION
Location: Vista Ballroom

Part 1: Determination of Polar Pesticides as Residual Impurities in Various Formulated Pesticide Matrices by LC-MS/MS

Chasity Love-Nkansah Ph.D., Chemist 5, Syngenta, Greensboro, NC, USA

A common threat for most agricultural companies is the possibility of cross-contamination of final products with residual impurities during manufacturing processes. Cross contamination of final products can result in substantial crop loss, possible regulatory violations, and significant damage to the company's reputation. The current practice for testing for residual impurities is to conduct targeted analysis for the active ingredient (s) in the preceding product. Currently, the analysis of polar pesticides are challenging and time-consuming due to them being highly soluble in water, their ionic nature, and various extraction difficulties.

For this presentation, we will show the application of a Waters Torus DEA column for the determination of underivatized polar pesticides in different formulated matrices and rinse water samples. This is an alternative

simpler method from the common practice of tedious FMOC derivatization. Waters Acquity UPLC H-Class coupled with a Xevo TQ MS was used for all of this work. The suggested protocol to activate and condition the Torus DEA column with a disodium EDTA solution was carried out to achieve better performance. We will show that that method achieved suitable ppm level detection, reproducible retention times for various pesticides, specificity in complex matrices, and linearity.

Part 2: Tips for Faster Sample Preparation

Jeremy Shia, Ph.D., Senior Product Manager Consumables Group, Waters Corporation, Milford, MA, USA

Jeremy will explore some recent work our scientific operations team has been working on to speed up your sample prep workflows. He will present data on one and two step workflows for the rapid removal of matrix interferences. Stop by our booth later for a personal demonstration of the cartridge in action with an interactive experiment

V-3 Monday, July 22, 2019, 12:15 to 1:15 pm

AGILENT TECHNOLOGIES

Location: Vista Ballroom

Part 1: Optimizing Sample Preparation in Pesticides Analysis for Cannabis

Jessica Westland, Agilent Technologies

Many U.S. state-regulated pesticides lists for cannabis can be analyzed exclusively by LC/MS/MS. Notable exceptions include California, Florida, and Nevada, where GC/MS/MS is also required. The states requiring GC/MS are expected to grow as more compounds and lower detection limits are required. In this work, the detection and quantitation of all LC-amenable pesticides and mycotoxins were reliably met by at least 50% of the current California legislative safety action limits in cannabis dried flower samples (limits of detection (LODs) range between 0.5 to 50 ppb; Malathion's LOD = 100 ppb). Forty-three GC-amenable pesticides regulated by the Bureau of Cannabis Control in California met the established limits of quantitation (LOQs) with the Agilent 8890 GC combined with an Agilent 7010B triple quadrupole GC/MS system. The Agilent standardized sample preparation procedure aligned with the Agilent multiplatform approach provides a rapid return on investment (ROI) and a stable foundation to meet current and future testing requirements.

Part 2: LC-MS/MS vs. LC-MS/MS & GC-MS/MS for the Certain Analysis of Pesticides in Cannabis Flower

Anthony Macherone, Agilent Technologies & The Johns Hopkins University School of Medicine

Pesticide analyses in cannabis flower is complicated because of the multitude of co-extractive endogenous chemicals and target analytes like pentachloronitrobenzene (PCNB) that are not amenable to electrospray ionization (ESI). To overcome this issue, it has been suggested to use negative atmospheric pressure chemical ionization liquid chromatography-tandem mass spectrometry (Ni-APCI LC-MS/MS) for compounds like PCNB to conform to a single analytical platform approach. However, this approach results in non-selective, non-linear precursor / product ion pairs that yield poor statistical regression coefficients and do not meet regulatory requirements in U.S. states like California. In this presentation we use using Ni-APCI LC-time-of-flight mass spectrometry (Ni-APCI LC-QTOF) to prove the correct ionization mechanism for PCNB and evaluate the appropriateness of this analytical technique for PCNB in terms of selectivity, sensitivity, and the fit of the coefficient of determination compared to GC-MS/MS methodologies.

V-4 Tuesday, July 23, 2019 7:00 to 8:15 am

BRUKER

Location: Vista Ballroom

Mass Spectrometric Solutions for Accurate Screening and Quantitation of Chemical Residues in Food and the Environment

Artem Filipenko, Ph.D., Bruker Scientific, Billerica, USA

With increasing demands for lower detection thresholds to cover hundreds of pesticides originating from numerous sample types, accurate and reliable pesticide screening is a critical and complex analytical task. To meet these challenging demands, Bruker has developed new UHPLC-QTOF and GC-APCI-QTOF based solutions that will be discussed at the seminar.

Fast and comprehensive full scan accurate mass screening and quantitation technique has become an excellent tool for food control and environmental analysis. This technique takes advantage of both targeted and non-targeted workflows, as well as data mining and retrospective analysis. The new TargetScreener HR 4.0 application kit is based on the Bruker QTOF platform. For maximum flexibility and depending on the application, TargetScreener can be operated in either UHPLC-QTOF or GC-APCI-QTOF configurations interchangeably. A central part of this solution is the new TASQ 2.1 Screening & Quantitation Software for rapid data processing that includes predeveloped analytical methods. Rigorously curated TargetScreener databases is pivotal for minimizing false positives and negatives and includes more than 3,000 entries relevant for food safety, environmental protection, and other screening and research applications.

V-5 Tuesday, July 23, 2019, 12:15 to 1:15 pm

LECO CORPORATION

Location: Vista Ballroom

LECO Corporation's NACRW Midsummer Classic, a Lunchtime Double Header ***"If you don't know where you're going, you might not get there."* Yogi Berra**

Part 1:

Bob Nelson, Woods Hole Oceanographic Institute

30-years pre-Deepwater Horizon oil spill an oil platform, Ixtoc-I, operating in the Bay of Campeche failed and spilled an estimated 3.5 M gal. into the Gulf of Mexico. This, second largest, lesser known oil spill has been investigated as a predictor for the 2010 Deepwater Horizon spill of 4.9 M gal. Using LECO's high resolution multidimensional technology to study Ixtoc-I samples, experts from Woods Hole Oceanographic Institute will tell us more on their findings into petroleum weathering across decadal time scales and how the data can be used to predict long-term fate of the crude oil released during the Deepwater Horizon disaster.

Part 2:

Todd Richards, LECO Corporation

In 2016 the EPA announced their ENTACT (EPA's Non-Target Analysis Collaborative Trial) program. The goals of this program were to identify which methodologies are appropriate for non-target detection and identification of common LC & GC amenable exposome compounds. This brief presentation will highlight on LECO's approach to non-target work, the strategies implemented and the tools that were created as a direct result of our participation in this trial.

V-6 Wednesday, July 24, 2019, 7:15 to 8:15 am

SCIEX

Location: Vista Ballroom

A New Generation in Chemical Residue Quantitation: Single Injection Analysis of 530 Mycotoxins, Metabolites and Other Emerging Masked Compounds

*Oscar G. Cabrices, Market Development Manager, Food/Beverage & Environmental Testing
SCIEX*

Success and growth of chemical residue testing laboratories often depends on their versatility to manage the diversity of samples received. High throughput quantitation without compromising sensitivity and robustness has always been the challenge for these labs due to the wide variety of processing steps and chromatography methods samples may require. In this seminar, we'll introduce the new SCIEX Triple Quad™ 5500+ QTRAP® Ready LC-MS/MS System designed for quantitative chemical residue analysis. A novel analytical workflow that utilizes rapid polarity switching for high throughput quantitation of 530 mycotoxins, metabolites and other emerging masked compounds in barley and corn extracts will be described.

V-7 Wednesday, July 24, 2019, 12:00 to 1:00 pm

PICKERING LABORATORIES

Location: Vista Ballroom

Part 1: Introduction of Onyx PCX – the newest addition to Pickering Laboratories' integrated family of post-column derivatization instruments, chemistry and support

Maria Ofitserova, PhD, Senior Research Chemist

Post-column derivatization technique is designed to enhance sensitivity and selectivity of detection. We demonstrate how Pickering Laboratories' Onyx PCX post-column derivatization system improves detection of variety of compounds, including Glyphosate, Mycotoxins, carbamates pesticides, and polyether antibiotics in complex matrices. Optimized instrumentation together with complete application support ensures ease of use, ruggedness and reliability of analysis.

Part 2: Analysis of Hemp Plant and Hemp-Containing Products using Post-Column Derivatization

Sareeta Nerkar, PhD, Research Chemist

The Hemp Farming Act of 2018 legalized hemp production and allowed sales of hemp plant and hemp-based CBD-containing products. With that came the need to test hemp products for contaminations as well as potency. We present post-column derivatization methods for analysis of mycotoxins and cannabinoids in dry hemp plant, full spectrum hemp oils, and full spectrum hemp edible products.

NACRW 2019



Join us at Booth 25 to discover our integrated portfolio of solutions

Join us at Booth 25 and learn about our highly integrated portfolio of separations and mass spectrometry products for the confident analysis of pesticides and other trace-level residues in complex matrices. Whether you perform routine or research analysis, Thermo Scientific workflow solutions are designed to improve the identification and quantitation of known and emerging contaminants in the areas of food and beverage, environmental, supplements, and more.

Featured product introduction

Experts will be at our booth to discuss our new Thermo Scientific™ Anionic Pesticides Explorer, a single, multi-analyte, robust, and sensitive “sample-to-result” IC-MS/MS workflow.

User meeting

Thursday, July 25 | 7:15–9:35 a.m. | Orchid 2

Current users, as well as those who are interested, are welcome to join us as we explore LC-MS and GC-MS technologies for dioxins and pesticide analysis.

Topics and presenters

Quantitative Analysis of Pesticides and PCBs in Fruit and Vegetables Using GC & LC Orbitrap

Dr. Jim Garvey, Department of Agriculture, Food and the Marine, Ireland

Use of HRAM LC-MS for Analysis of Pesticide Residues in the European Union

Professor Amadeo Fernandez-Alba, Universidad de Almería, Department of Agronomy, Spain

Thermo Scientific™ Anionic Pesticides Explorer

Ed George, Thermo Fisher Scientific

Thermo Scientific™ Pesticide Explorer

Charles Yang, Thermo Fisher Scientific

Thermo Scientific™ Orbitrap ID-X Tribrid™ MS with Thermo Scientific™ AcquireX intelligent MS™ Data Acquisition Strategy: Pesticides Quantitation and Screening

Natasa Kalli, Thermo Fisher Scientific

Dioxin Analysis in Food with the Thermo Scientific™ TSQ™ 9000 GC-MS/MS and Advanced Electron Ionization

Tim Anderson, Thermo Fisher Scientific

Register onsite at **Booth 25**

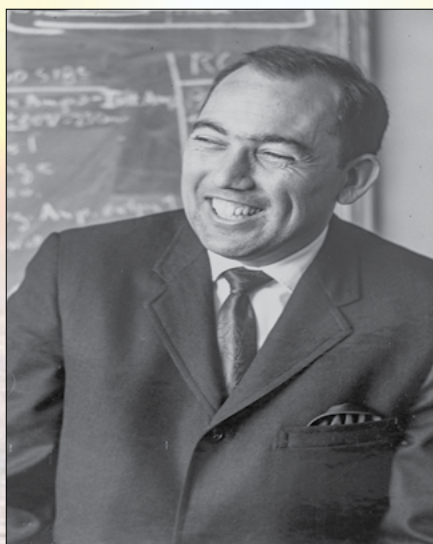
2019 NACRW EXCELLENCE AWARD

PRESENTED TO

Michael S. Story

Mike Story earned a B.S. Degree in Chemistry from the University of California, Berkeley in 1959 with an emphasis on physical chemistry. After fulfilling his military service and getting married he was involved for five years in material science research including planning, equipping, and establishing analytical methods for an instrumentation laboratory in the Microwave Tube Division of Litton Industries. He was then hired at Electronics Associates Inc., an analog computer manufacturer, by Dr. Robert Finnigan to help develop a quadrupole mass filter system for use as a residual gas analyzer (RGA). While there, Story built the first laboratory mass spectrometer based on quadrupole technology and offered for commercial sale. In 1967 he joined Bob Finnigan, Bill Fies and Dick Hein in Palo Alto CA to found Finnigan Corporation. For the next 34 years Mike participated in all phases of mass spectrometer research, development, and commercialization. He co-developed 4 mass spectrometer systems, organized a "skunkworks" in the research department to initiate the design, development, production and sale of first of its kind, triple quadrupole mass spectrometers patterned after the work of Enke and Yost, selected and managed the long term academic relationships of university consultants to commercialize new technology, had yearlong expatriate assignments in Germany and the UK integrating R&D capabilities of acquired companies, spent two years establishing a sales, service, and applications startup in Japan, and upon returning in the mid 80's, established and staffed an analytical biochemistry group to focus the companies instrument development on the emerging biological market. Among the many positions held were: Director of R&D of Finnigan Corp. and VP for Mass Spectrometry of Thermo-Finnigan.

He is a charter member, and has twice served on the Board, of the American Society for Mass Spectrometry and served for 12 years on National Academy of Science Advisory committees on detection of explosives in commercial aviation. He holds numerous instrumental patents, authored two chapters, and given presentations internationally on mass spectrometry instrumentation. He retired in 2001 with his wife, three sons, their wives, and 8 grandchildren who all live nearby.



1967



2005

The title of Mr. Story's presentation is:

**"How Environmental Analysis Saved a Company and a New Kind of Mass Spectrometer
Formation of Finnigan Corporation: A Personal Reflection"**

MEETING PROGRAM

Sunday, July 21, 2019

noon-6:00 pm	Registration	Orchid Foyer
1:00-5:00 pm	Exhibitor Setup	Royal Palm Ballroom
1:00 - 2:30 pm	NACRW Reference Materials Working Group <i>Develop processes and systems that will improve knowledge on the availability, use and stability of chemical residue reference materials and standard mixes. Identify needs and promote the development of chemical residue reference materials.</i>	Orchid 1-2
2:45 - 4:15 pm	NACRW Veterinary Drugs Working Group <i>Identify and recommend multi-residue veterinary drug methods that will meet the needs of government regulators.</i>	Orchid 1-2
4:30 - 6:00 pm	PERFORM Working Group Proficient, Equivalent, Reliable, Flexible, Officially Recognized Method <i>Develop a process that demonstrates equivalent performance of a user method to the performance required for Official methods.</i>	Orchid 1-2
3:00-6:00 pm	Poster Board Set Up	Royal Palm Ballroom
6:15-7:15 pm	Shimadzu Scientific Instruments, Inc. Vendor Seminar	Vista Ballroom, 1st level
7:30-9:30 pm	Welcome Reception	Royal Palm Ballroom

Monday, July 22, 2019

7:30 am-5:00 pm	Registration	Orchid Foyer
7:00-10:00 am	Poster Board Set Up	Royal Palm Ballroom
7:15-7:45 am	Moderator and Volunteer Training	Orchid Ballroom
7:15-8:15 am	Waters Corporation Vendor Seminar	Vista Ballroom, 1st level
7:30-8:15 am	Early Morning Coffee	Orchid Foyer
8:30-8:40 am	Opening Remarks Sherry Garris, Chair, NACRW Board of Directors	Orchid Ballroom
8:40-8:45 am	NACRW Excellence Award Presentation and Keynote Address Introduction and Presentation of NACRW Excellence Award Ken Kise, 2019 NACRW President	
8:45-9:30 am	Presentation by Excellence Award Winner Michael S. Story, Co-Founder, Finnigan Corporation, Los Gatos, CA A-1 How Environmental Analysis Saved a Company and a New Kind of Mass Spectrometer Formation of Finnigan Corporation: A Personal Reflection	
9:30-10:45 am	SESSION 1: Single Residue and Small Scale Multiresidue Methods Co-Chairs: Katherine Carlos and Lynda Podhorniak	Orchid Ballroom
9:30-9:50 am	O-1 A Versatile Method for the Analysis of Glyphosate in Raw Agricultural Commodities, Foods, and Other Complex Matrices using LC-MS/MS Satya Avula, Eurofins Food Integrity & Innovation, Brownsburg, IN, USA	
9:55-10:15 am	O-2 The Enzymatic paper-on-a-chip concept: Towards the On-Site Organophosphate and Carbamate Pesticides Detection in Fruits and Vegetables Aristeidis Tsagkaris, University Of Chemistry And Technology, Prague, Czech Republic	

2019 - 56th ANNUAL NORTH AMERICAN CHEMICAL RESIDUE WORKSHOP

10:20-10:40 am	O-3	Analysis of Ocean Dump Site Samples for Single Class Persistent Organic Pollutants (POPs) Using Comprehensive Gas Chromatography Coupled with High Resolution Mass Spectrometry Robert Nelson, Woods Hole Oceanographic Institution, Woods Hole, MA, USA	
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		Agilent Vendor Seminar	Vista Ballroom, 1st level
1:30-3:10 pm		SESSION 2: Method Validation and Verification Co-Chairs: Paul Reibach and Kate Mastovska	Orchid Ballroom
1:30-1:50 pm	O-4	Method Validation vs. Verifications – A Discussion Rebecca Smith, Smithers Viscient, Wareham, MA, USA	
1:55-2:15 pm	O-5	Development and Validation of a GC-HRAM Method for the Quantitation of Pesticide Residues in Fruit and Vegetables Jim Garvey, The Pesticide Control Laboratory, Celbridge, Co. Kildare, Ireland	
2:20-2:40 pm	O-6	Focus on Analytical Excellence: The Latest on AOAC Programs, Services, and Processes Deborah Mckenzie, AOAC International, Rockville, MD, USA	
2:45-3:05 pm	O-7	Determination of Cannabinoids in Plant Materials, Oils and Concentrates: Single Laboratory Validation for AOAC First Action Official Method Consideration Lukas Vaclavik, Eurofins Food Integrity & Innovation, Harrogate, United Kingdom	
3:10-3:55 pm		BREAK (Exhibition & Posters)	Royal Palm Ballroom
3:55-5:45 pm		SESSION 3: Analysis of Cannabis Co-Chairs: Julie Kowalski and Jessica Krank	Orchid Ballroom
3:55-4:00 pm	O-8	Evaluation of Sample Preparation Techniques for Cannabis and Cannabis Products Kelsey Cagle, Pennsylvania State University, State College, PA, USA	
4:05-4:25 pm	O-9	Pesticide Residue Analysis in Cannabis: Extraction and Cleanup Strategies for LC-MS/MS and GC-MS/MS Rebecca Stevens, ProVerde Laboratories, Milford, MA, USA	
4:30-4:50 pm	O-10	Streamlined Pesticides Multi-Residue Methods for Cannabis and Various Plant Vegetation Matrices David Blais, Health Canada, Ottawa, Ontario, Canada	
4:55-5:15 pm	O-11	Important Considerations for Proper Determination of Limits of Detection and Quantitation Paul Winkler, SCIEX, Golden, CO, USA	
5:20-5:40 pm	O-12	The Scientific Rationale for Multiplatform Analytical Technologies for the Analysis of Certain Pesticides in Dry Cannabis Flower Anthony Macherone, Agilent & Johns Hopkins, Wilmington, DE, USA	
6:30 pm →		SOCIAL EVENT – Naples Grande	Sunset Veranda/Vista Ballroom

2019 - 56th ANNUAL NORTH AMERICAN CHEMICAL RESIDUE WORKSHOP

Tuesday, July 23, 2019

7:30 am-5:00 pm	Registration	Orchid Foyer
7:30 am-4:00 pm	Exhibition & Posters	Royal Palm Ballroom
7:00-8:15 am	Bruker Vendor Seminar	Vista Ballroom, 1st level
7:30-8:15 am	Early Morning Coffee	Royal Palm Ballroom
8:45-10:45 am	SESSION 4: Multi-residue Methods for Pesticides and Related Contaminants Co-Chairs: Martin Dusek and Jon Wong	Orchid Ballroom
8:45-9:10 am	O-13 Dealing with Matrix in the Analysis of Pesticide Residues in Atmospheric Particles, Bees and Bee Products, and Botanical Products by Liquid Chromatography-Tandem Mass Spectrometry Renata Raina-Fulton, University of Regina, Department of Chemistry & Biochemistry, Regina, Saskatchewan, Canada	
9:15-9:40 am	O-14 Improved High Throughput Suspect Screening Analysis of Pesticides using a LC/Q-TOF and Novel Software Tool Karen Yannell, Agilent Technologies, Santa Clara, CA, USA	
9:45-10:10 am	O-15 The Renaissance of Supercritical Fluid Chromatography in Food Pesticide Residue Analysis? Amadeo Rodriguez Fernandez-Alba, EURL-FV University of Almería, La Cañada de San Urbano, Almeria, Spain	
10:15-10:40 am	O-16 New Efficient Approach for the NL-Acetone Extraction Method for Pesticide Residue Analysis by LC- and GC-MS/MS Ionara Pizzuttia, Federal University of Santa Maria, Santa Maria, Brazil	
10:45-noon	Exhibition and Poster Opening	Royal Palm Ballroom
11:00-noon	Poster Session B (authors present for even #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, 1st level
1:30-3:10 pm	SESSION 5: Novel and Emerging Contaminants Co-Chairs: Susie Genualdi and Neely Belai	Orchid Ballroom
1:30-1:50 pm	O-17 Challenges in the Analysis of Perfluoroalkyl Substances in Food Matrices by LC-MS/MS at sub-ppb Concentration Bjorn Berendsen, Wageningen Food Safety Research, Wageningen, Netherlands	
1:55-2:15 pm	O-18 Identification and Determination of BFRs in Food Contact articles and Food Rafael Paseiro Cerrato, U.S. Food and Drug Administration, College Park, MD, USA	
2:20-2:40 pm	O-19 Quantitative Analysis of Naturally Occurring Compounds Regulated under California's Safe Drinking Water and Toxic Enforcement (Prop 65) Act by SPME-HS Wiley Hall, Safe Food Alliance, Kingsburg, CA, USA	
2:45-3:05 pm	O-20 Improved Extraction and Cleanup Prior to HPLC Determination of Glyphosate in Food Samples Sareeta Nerkar, Pickering Laboratories, Inc., Mountain View, CA, USA	

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3:10-3:55 pm	BREAK (Exhibition & Posters)	Royal Palm Ballroom
3:55-5:00 pm	SESSION 6: Pesticide Residue Forum Moderators: Walter Hammack, Simon Hird, Brian Eitzer	Orchid Ballroom
5:05-6:00 pm	NACRW Organizing Committee Meeting	Orchid Ballroom <i>open to all attendees</i>
6:00-10:30 pm	Shuttle service to and from Mercato (Blue Martini) <i>One shuttle scheduled to run every 15 minutes</i>	Outside - Hotel Main Entrance

Wednesday, July 24, 2019

7:30 am-noon	Registration	Orchid Foyer
7:30 am-noon	Exhibition & Posters	Royal Palm Ballroom
7:15-8:15 am	SCIEX Vendor Seminar	Vista Ballroom, 1st level
7:30-8:15 am	Early Morning Coffee	Royal Palm Ballroom
8:30-10:45 am	SESSION 7: State/Federal Lab Updates Co-Chairs: Yoko Johnson and Ken Kise	Orchid Ballroom
8:30-8:55 am	O-21 Colorado Department of Agriculture Laboratory Update Jessica Krank, Colorado Department Of Agriculture, Denver, CO, USA	
9:00-9:25 am	O-22 nDATA workflow for Screening Pesticides in Fruits and Vegetables Jon Wong, U.S. Food and Drug Administration, College Mark, MD, USA	
9:30-9:55 am	O-23 A High Resolution Mass Spectrometry (HRMS) Method for More 1000 Pesticides and other Poisons: The Method and Madness Marc Engel, Florida Department of Agriculture and Consumer Services, Tallahassee, FL, USA	
10:00-10:25 am	O-24 Analysis of Sodium Fluoroacetate (1080) and Dicamba using the Gerstel Multi Purpose Sampler DHS and Twister® capabilities Jean-Paul Schirlé-Keller, Minnesota Department Of Agriculture, Saint Paul, MN, USA	
10:30-10:45 am	Student Scholarship Presentations	Orchid Ballroom
10:45-noon	BREAK (Exhibition & Posters)	Royal Palm Ballroom
12:00-1:00 pm	Pickering Laboratories Vendor Seminar	Vista Ballroom, 1 st level
1:05-2:45 pm	SESSION 8: Trends in Veterinary Drug Residue Control Co-Chairs: Sherri Turnipseed and Eric Verdon	Orchid Ballroom
1:05-1:25 pm	O-25 Multiresidue Method for Analysis of Veterinary Drug Residue In Meat By LC-HRMS - Screening and/or Quantitation? Dominique Hurtaud Pessel, Anses, Laboratory of Fougères, Fougères, France	
1:30-1:50 pm	O-26 Development and Validation of a QuEChERS Mega-Method for the Analysis of Pesticides, Veterinary Drugs, and Environmental Contaminants in Fish and Meat Steven Lehotay, USDA Agricultural Research Service, Wyndmoor, PA, USA	
1:55-2:15 pm	O-27 Occurrence of Residues of Veterinary Antibiotics in Water, Sediment and Trout Tissue (Oncorhynchus mykiss) I n the South area of Titicaca Lake,PERU Franz Vilca, Universidad Nacional De Moquegua, Moquegua - Ilo, PERU	
2:20-2:40 pm	O-28 Occurrence of Antibiotics and Veterinary Drug Residues in Wildlife Ovokeroye Abafe, Agricultural Research Council, Pretoria, Gauteng, South Africa	

2019 - 56th ANNUAL NORTH AMERICAN CHEMICAL RESIDUE WORKSHOP

2:45-3:15 pm	BREAK	Orchid Foyer
3:15-4:55 pm	SESSION 9: Emerging Topics in Trace Level Analysis Co-Chairs: Marc Engel	Orchid Ballroom
3:15-3:35 pm	O-29 The Need for Safety and Authenticity Testing Methods for Natural Colors Joe Konschnik, Restek Corporation, Bellefonte, PA, USA	
3:40-4:00 pm	O-30 Perfluoroalkyl compounds in Milk and Maple Syrup Robert Sheridan, New York State Department Of Agriculture, Albany, NY, USA	
4:05-4:25 pm	O-31 Advances and Data Gaps in Analytical Discovery, Detection, and Quantitation of Algal Toxins Keith Loftin, U.S. Geological Survey, Lawrence, KS, USA	
4:30-4:50 pm	O-32 Emerging Algal Toxins in California: Responding to Known Threats, Preparing for the Future Raphael Kudela, University Of California Santa Cruz, Santa Cruz, CA, USA	
4:55-5:10 pm	Poster Awards and Closing	Orchid Ballroom

Thursday, July 25, 2019

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

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ORAL PRESENTATION ABSTRACTS

A-1 How Environmental Analysis Saved a Company and a New Kind of Mass Spectrometer Formation of Finnigan Corporation: A Personal Reflection

Michael S. Story, Co-Founder, **Finnigan Corporation**, Los Gatos, CA

At the time Finnigan Corporation (Palo Alto, CA) was formed in 1967, mass spectrometry was an integral tool for organic analysis. Overwhelmingly, mass spectrometers were sector field instruments requiring significant space, expensive to purchase, complex to operate, and with low sample throughput. Although gas chromatographs were an obvious inlet/sample preparation device, GC/MS was practiced in only a select few laboratories and most notably for biological applications. Data were single scans recorded on fast chart recorders requiring each scan to be manually measured for mass assignments and interpreted by experts.

The future founders of Finnigan Corporation had already considerable experience with quadrupole mass filter technology for use in residual gas analysis, mostly for space and semiconductor research. This experience harnessed by the entrepreneurial spirit of Bob Finnigan, led a small group to believe they could put GC-MS-DS in the hands of the chemist by developing a quadrupole-based system. Quadrupole technology was small, less expensive to build, rapid scanning and, very importantly, easily computer controlled. In Silicon Valley terms, it was an “enabling technology” and it would later be shown to be disruptive as well. The emerging needs of environmental analysis provoked by Rachel Carson’s “Silent Spring” greatly enhanced our chance of survival.

Bob Finnigan, Bill Fies, Richard Hein, and Mike Story left their employers for an uncertain future. Within 4 years of formation, a gas chromatograph-quadrupole mass spectrometer-data system that could be operated by a trained bench chemist was commercially offered. The “startup’s” success required choosing which scientific, engineering, financial and market problems to solve. In order to do this, alliances with Universities were made, a second independent company was formed, researchers in academic and industrial laboratories “took a chance” on an unproven technology and someone from the government who was really “here to help” was found. The personal “Silicon Valley” networking connections, interrelated circumstances, role of environmental analytical needs, and developmental research that allowed the Company to survive and prosper will be described.

O-1 A Versatile Method for the Analysis of Glyphosate in Raw Agricultural Commodities, Foods, and Other Complex Matrices using LC-MS/MS

Satya G.C. Avula,¹ John P. Zulkoski,² Lukas Vaclavik,³ and Katerina Mastovska;²

¹**Eurofins Food Integrity & Innovation**, 671 S. Meridian Rd., Greenfield, IN 46140, USA; SatyaAvula@eurofinsUS.com;

²Eurofins Food Integrity & Innovation, 3301 Kinsman Blvd., Madison, WI 53704, USA;

³Eurofins Food Integrity & Innovation, Otley Road, Harrogate, North Yorkshire, HG3 1PY, United Kingdom

A versatile, high-throughput and sensitive method was developed for the analysis of glyphosate [N-(phosphomethyl) glycine] and its metabolite AMPA (aminomethylphosphoric acid) using ultra-high performance liquid chromatography coupled to tandem mass spectrometry (LC-MS/MS). The applied method enables quantitation in various raw and processed agricultural products (including juices, grains, high fiber products, corn starch, legumes, nuts and seeds) and complex matrices such as botanicals. Aqueous extracts from the samples are derivatized with FMOC (fluorenylmethyloxycarbonyl chloride) and are further cleaned up by solid phase extraction (SPE). Stable-isotope labeled internal standards are used for each analyte to correct for instrument response and losses during sample preparation. The sample size, extraction solvents, derivatization conditions and the cleanup procedure were thoroughly optimized and the method performance was evaluated according to the SANTE/11813/2017 guidelines for a target limit of quantitation (LOQ) of 0.01 – 0.1 mg/kg (matrix dependent). The validated method provides a single analytical work flow for the routine analysis of glyphosate and AMPA in a wide range of matrices.

O-2 The enzymatic paper-on-a-chip concept: Towards the on-site organophosphate and carbamate pesticides detection in fruits and vegetables

Aristeidis Tsagkaris, Daniel Filippini, J.-Pablo Salvador, M.-Pilar Marco, Jana Pulkrabova, and Jana Hajslova

University Of Chemistry and Technology, Prague, Czech Republic

Lab-on-a-chip (LOC) devices is a concept of increasing popularity because it provides miniaturization, integrated sample handling and low-cost measurement. Although the determination of organophosphate and carbamate pesticides requires expensive and time-consuming chromatographic methods, rapid screening can be achieved based on the principle that organophosphates and carbamates inhibit acetylcholinesterase (AChE). In-vitro, AChE can hydrolyze certain colorless substrates to color products. However in the presence of an inhibitor, AChE is inhibited dependently on a pesticide concentration and this inhibition can be correlated to a color intensity decrease. To this end, we developed an interdisciplinary analytical platform for organophosphates and carbamates detection. To begin with, AChE was physically adsorbed on Whatman paper. Then, two AChE strips were incorporated in a prototype LOC creating a so called enzymatic paper-on-a-chip (EPOC) device. The EPOC device provided integrated samples and substrates loading using silicon tubes as a pumping element eliminating the pipetting need. To monitor the color development on AChE strips, the EPOC device was placed in an in-house 3D-printed smartphone reader. Paper assay images were captured using the OpenCamera application and image data processing was performed in MatLab. Finally, the developed platform was successfully used to quantitatively detect carbofuran in apple extracts at sub-ppm level. Despite being a restricted pesticide in the EU, carbofuran was recorded among the pesticides with frequent MRL exceedances in the latest EFSA report indicating the need for rapid screening methods. All in all, this fast, simple and cost-effective approach can be a paradigm shift towards the on-site chemical residues detection.

O-3 Analysis of Ocean Dump Site Samples for Single Class Persistent Organic Pollutants (POPs) Using Comprehensive Gas Chromatography Coupled with High Resolution Mass Spectrometry

Robert K. Nelson,¹ Veronika Kivenson,² Karin L. Lemkau,² Oscar Pizarro,⁴ Dana R. Yoerger,¹ Carl Kaiser,¹ Catherine Carmichael,¹ Blair G. Paul,² Christopher M. Reddy,¹ and David L. Valentine²

¹**Woods Hole Oceanographic Institution**, Woods Hole, Massachusetts 02453, USA; rnelson@whoi.edu

²University of California, Santa Barbara, California 93106, USA

³University of California, Santa Barbara, California 93106, USA

⁴Australian Centre for Field Robotics, University of Sydney, Sydney 2006, Australia

Using the autonomous underwater vehicle (AUV) Sentry and the remotely operated vehicle (ROV) Jason, an abandoned industrial waste deep-sea dump site located in the Southern California Bight, was mapped, inspected, and sampled. A technical report by Chartrand et al., states this area may contain 336,000–504,000 barrels of acid sludge waste from the production of persistent organic pesticides (POPs), in addition to various other waste streams. Montrose Chemical Corporation (MCC) was the largest global supplier of DDT, manufacturing ~ 800,000 tons at their Los Angeles County plant from 1947 to 1982. Offshore disposal of manufacturing byproducts resulted in at-sea dumping of an estimated 2000–3000 barrels/month, ~1 million gallons of waste per year, from 1947 to 1961. Ocean dumping practices were prescribed by California Department of Fish and Game, US Coast Guard, and Los Angeles Harbor Department, specifying waste hauling companies dump containerized waste at designated locations in the basins off Southern California, ~16 km offshore; however, short-dumping closer to shore was common practice. Discarded waste containers litter this region and the leaked contents of these containers are abundant in the suboxic benthic environment around this site, thus providing an entry point into the marine food web. Following ROV retrieval, sediment cores were processed, stored and subsequently analyzed at the Woods Hole Oceanographic Institution. Here we describe the analysis of sediment cores from the Southern California Bight for POPs by comprehensive two-dimensional gas chromatography coupled with high resolution time of flight mass spectrometry.

O-4 Method Validation vs. Verifications – A Discussion

Rebecca J. Smith¹, and Paul Reibach¹

¹Smithers Viscient, 790 Main Street; Wareham, MA 02571, USA; rsmith@smithers.com

Developing and validating a robust, regulatory compliant analytical method can be an overwhelming process. Single analyte residue methods are often developed and utilized outside of enforcement laboratories, or when a pesticide residue cannot be determined using a multi-residue method. These methods are ideally developed to include the determination of all analytes included in the residue definition of a particular substance. When a lab finds themselves in a position where they need to develop or adopt and validate an analytical method, a number of questions must be considered. Does the method validation need to be done in accordance to GLPs? What is the difference between a method “validation” and a method “verification”? Is there a particular guideline or guidance document that should be followed? How do I ensure my method is robust? Do I need to conduct a formal Environmental Chemistry Method (ECM) or have an Independent Lab Validation (ILV) performed? What about method transfers to or from the lab? This presentation will focus on these common questions. In addition, the differences between validations, verifications and method transfers will be examined, as well as what particular elements of a method validation will ensure proper method performance and reproducibility.

O-5 Development and validation of a GC-HRAM method for the quantitation of pesticide residues in fruit and vegetables

Jim Garvey, Elaine Devaney, Teresa King and Ross Kilduff

The Pesticide Control Laboratory, Celbridge, Co. Kildare, Ireland

With the increasing demand on pesticide residue laboratories to increase their scope of analysis high resolution accurate mass (HRAM) systems have found increasing popularity in this area. The systems have the advantage of much more reliable confirmation due to the mass accuracy achieved. To date much of the work involving these systems has revolved around developing screening methods and little has been done on use of these systems for quantitative methods. Here we describe the development and validation of a quantitative method for the analysis of 167 pesticide residues and PCB's in samples of fruit and vegetables according to the protocol described in EU Document No. SANTE/2017/11813. The determination method involves analysis using a GC QExactive orbitrap in full scan mode using EI ionisation. The samples were then extracted using the standard miniLuke method. After extraction with acetone/dichloromethane/petroleum ether 40-60oC a solvent exchange into ethyl acetate is carried out. Recovery work was carried out in cucumber, lemon and broccoli.

The results show that the default MRL of 10ppb can be achieved for more than 93% of the pesticides studied. Mass accuracy, ion ratio and matrix effect studies show that the method is robust and provides a viable alternative to triple quadrupole mass spectrometer systems for the quantitation of pesticide residues in fruit and vegetable samples. The increased confidence provided by the HRAM system gives results with very high reliability.

O-6 Focus on Analytical Excellence: The Latest on AOAC Programs, Services, and Processes

Deborah McKenzie¹

¹AOAC INTERNATIONAL, 2275 Research Boulevard, Suite 300; Rockville, MD 20850, USA; dmckenzie@aoac.org

When looking to implement a method in the laboratory, do you ever wonder how those AOAC methods became AOAC methods? What are AOAC method related programs and what are the difference between them? What are the processes for methods under review in AOAC and how do they become available for your laboratory's consideration? For almost 30 years, the overall methods process was consistent and well known. To address the need for emerging technologies and methods development and validation on an expedited timeline led an evolution in AOAC that informed initial changes in its method programs and processes in 2007. Since this time, there has been continued growth and

evolution of the programs; however, as part of this continued evolution, the processes and requirements for methods going through AOAC becomes less obvious and requires continued communication and education. As part of the latest on the methods related processes, AOAC has implemented its new Analytical Solutions Forum as part of the processes that lead to AOAC's programs and services, providing for a complete quality system. These newest evolution of processes for methods are part of a new and ongoing effort by AOAC to fulfill its mission and strategic goal of analytical excellence, continuing to provide fit-for-purpose methods and solutions.

O-7 Determination of Cannabinoids in Plant Materials, Oils and Concentrates: Single Laboratory Validation for AOAC First Action Official Method Consideration

Lukas Vaclavik¹, Frantisek Benes², Marie Fenclova², Jiri Hricko², Ales Krmela², Veronika Svobodova², Jana Hajslova² and Katerina Mastovska³

¹Eurofins Food Integrity & Innovation, Otley Road, Harrogate, North Yorkshire, HG3 1PY, UK; LukasVaclavik@eurofins.co.uk;

²University of Chemistry and Technology Prague, Technicka 5, 160 00 Prague, Czech Republic;

³Eurofins Food Integrity & Innovation, 3301 Kinsman Boulevard, Madison, WI 57304, USA

This presentation describes a single-laboratory validation (SLV) of a liquid chromatography–diode array detection (LC–DAD) method for quantification of twelve major cannabinoids in Cannabis dried plant materials, oils and concentrates. The method met AOAC Standard Method Performance Requirements (SMPRs) for quantitative analysis of cannabinoids in Cannabis concentrates and Cannabis dried plant materials. The limits of quantification (LOQs) were in the range 0.003 - 0.10 % (w/w), depending on the analyte and matrix. Spike recoveries were between 96.7 - 101.3% with relative standard deviations (RSDs) ≤ 2.3%. Precision expressed as repeatability (RSDr) and intermediate precision (RSDINT) was within 0.3 - 4.8 % and 1.1 - 5.1 %, respectively. The chromatographic separation conditions used in this versatile method are compatible with both DAD-UV and mass spectrometric detection. During method validation, high-resolution quadrupole-time-of-flight mass spectrometer (Q-TOFMS) was employed as a secondary detector (connected in series to the LC-DAD instrument) to provide high confidence identification of target analytes. The obtained results demonstrate excellent performance of the method in quantitative analysis of important cannabinoids in dried plants, concentrates and oils.

O-8 Evaluation of Sample Preparation Techniques for Cannabis and Cannabis Products

Kelsey Cagle¹, Jessica Westland², and Frank L. Dorman¹

¹The Pennsylvania State University, University Park, PA 16801, USA; kzc225@psu.edu; ²Agilent Technologies, Inc., Wilmington, DE 19808, USA

Sample preparation is an essential part of method development that is critical to successful analytical measurements. In short, the goal for organics analysis is to reduce and simplify the complex sample matrices to a form that is applicable for instrumental analysis without bias to potential target and non-target compounds. With cannabis and cannabis products, however, the analyst is faced with a very challenging matrix and targets that may range from trace level through percent level thus placing considerable demands on the sample preparation techniques.

Recently, sample preparation for cannabis and cannabis products has moved towards QuEChERS-like procedures due to their high throughput and lower costs. QuEChERS-like procedures provide a more generic approach that is well suited for a variety of products and also provides sample cleanup that increases compatibility for both GC and LC separations. QuEChERS' appeal to cannabis work is its ability to be modified in order to deal with matrix effects commonly associated with cannabis.

This presentation will discuss the results from a consolidated sample preparation technique for cannabis and cannabis products that will allow for the analysis of incurred pesticides residues and potency evaluations, ideally from the same sample preparation process. Data from both GC-MS/MS and HPLC will be shown to demonstrate the effectiveness of this approach.

O-9 Pesticide Residue Analysis in Cannabis: Extraction and Cleanup Strategies for LC-MS/MS and GC-MS/MS

Rebecca Stevens,¹ Christopher J. Hudalla,¹ Michael S. Young,² Marian Twohig,² Kim Tran,² Kari Organtini,²

¹ProVerde Laboratories, Milford, MA, USA

²Waters Corporation, Milford, MA, USA

Multi-residue pesticide methods for cannabis must provide reliable and robust quantitative analysis to meet regulatory limits for the entire list of required analytes. The sensitivity and specificity offered by LC-MS/MS is suitable for the determination of most pesticide residues in cannabis. However, not all pesticides ionize efficiently by the electrospray or atmospheric pressure chemical ionization modes typically used for LC-MS. Therefore, only the most sensitive (and most costly) LC-MS instruments can be used to quantify those poorly ionized pesticides although with a higher potential for matrix interference and suppression. Fortunately, most of the pesticides with very low LC-MS sensitivity have much better response using GC-MS analysis. Therefore, the application of both LC-MS/MS and GC-MS/MS will achieve the lowest possible detection limits for the widest range of pesticide residues. As important as the instrumental considerations for pesticide residue analysis in cannabis are the considerations for sample preparation. Various extraction procedures were investigated including QuEChERS and direct solvent extraction. For cleanup, both dispersive and pass-through types of SPE were evaluated. In summary, methods were developed for multi-residue pesticide analysis in cannabis using both LC-MS/MS and GC-MS/MS after optimized extraction and cleanup. Method performance was evaluated by assessing recovery, matrix suppression, linearity, and sensitivity.

O-10 Streamlined Pesticides Multi-Residue Methods for Cannabis and Various Plant Vegetation Matrices

Michel Blais, Mathieu Gagnon, Tyler McRitchie, Kim Montsion, Josee Tully, Neil Snider, David R. Blais

Laboratory Services, Pest Management Regulatory Agency, **Health Canada**, Ottawa, Ontario, K1A 0C6, Canada; David. Blais@Canada.ca

Quantitative analytical methods were streamlined and miniaturized to accommodate both dry and high water content vegetation samples, such as cannabis leaves, dried cannabis and cannabis oil. The quantity of vegetation material has been reduced to 4 g for high moisture content samples or 2 g for low moisture samples, followed an acetonitrile extraction, NaCl dispersive C18 clean-up, solid phase extraction cleanup and analysis by High-Res LC-MS, HPLC-MS/MS and GC-MS/MS. The cannabis oil method was developed to accommodate the lipid content of the matrix with a 50:50 acetonitrile:acetone extraction, QuEChERS cleanup, and analysis through High-Res LC-MS, HPLC-MS/MS and GC-MS/MS. Validation at spiking levels as low as 0.01 µg/g was successful, with the majority of the 350 pesticide residues showing recoveries within the acceptable range of 70-130%. These methods are being used for unannounced compliance and enforcement inspections across Canada, including cannabis licensed producers.

O-11 Important Considerations for Proper Determination of Limits of Detection and Quantitation

Paul Winkler¹, Diana Tran¹, Simon Roberts¹, Karl Oetjen¹, Chris Borton¹, Ty Garber²

¹SCIEX, 500 Old Connecticut Path, Framingham, MA 01701, USA; paul.winkler@sciex.com

²Phenova, Arvada, USA

An important criterion for pesticide testing in cannabis is the action limit that is specified in the current regulation. This is particularly important for cannabis analyses because a failure will result in the destruction of an expensive product or a false negative can result in contamination in concentrates and edibles later in the process which will also result in the destruction of product. Two important parameters for the accurate determination of the action Limit are the Limit of Detection (LOD) and Limit of Quantitation (LOQ). There are many different techniques that may be used to determine the LOD and LOQ. One common method is to analyze a matrix sample spiked at a concentration and analyzing seven replicates. The standard deviation of these replicates is then used to calculate either a LOD or a LOQ, depending on the spiking level. There is a serious downside to this procedure, however, that may lead to inaccurate and unrealistic numbers for the LOD and LOQ. The number that is calculated may be a concentration that is not actually measurable,

and it is important to repeat a set of experiments at or near the calculated LOD or LOQ so that it may be demonstrated that the calculated LOD or LOQ is real. The data that will be presented here will be from an LOD and LOQ experiment and the calculated results will be compared to matrix samples that were spiked at concentrations near the calculated values. The results of this comparison will be discussed and the importance of requiring the verification spike will be demonstrated.

O-12 The Scientific Rationale for Multiplatform Analytical Technologies for the Analysis of Certain Pesticides in Dry Cannabis Flower

Anthony Macherone^{1,2}

¹ **Johns Hopkins University** School of Medicine, Department of Biological Chemistry, 733 N Broadway; Baltimore, MD 21205, USA; amacher1@jhmi.edu

² **Agilent Technologies**, 2850 Centerville Road; Wilmington, DE 19808, USA

In U.S. states and Canada where cannabis has been legalized, regulatory entities require a panel of chemical and biological tests to be performed to assure safety of the products. Of the required assays, residual pesticide analysis is arguably the most challenging. The reason for this is the complexity of the cannabis flower that synthesizes myriad endogenous chemicals. It is not unusual for today's cannabis to contain 20% - 30% Δ^9 -tetrahydrocannabinol (THC) by dry weight. In contrast, residual pesticides are typically measured in the 10 – 1000 ng/g (ppb) range. Tandem quadrupole mass spectrometry (MS/MS) is the primary analytical platform for this analysis due to its thorough mitigation of chemical noise. Notwithstanding the power of MS/MS, there are many cases where isobaric interferences affect quantitative results and therefore specificity becomes as, or more important than sensitivity. In this study, we used liquid chromatography, and gas chromatography time-of-flight mass spectrometry to evaluate the selectivity of a model pesticide commonly found in regulatory target lists. The LC system employed negative ion-atmospheric pressure chemical ionization (Ni-APCI), and the GC system employed electron ionization (EI). Through this work, we demonstrated that EI precursor ion / product ion pairs are highly specific derivatives of the parent molecule while the Ni-APCI precursor ion is a non-specific transformation product resulting from a complex ionization mechanism. In this latter case, all precursor ion / product ion pairs are therefore not specific to the parent molecule and increase the probability of falsely positive or negative results.

O-13 Dealing with Matrix in the Analysis of Pesticide Residues in Atmospheric Particles, Bees and Bee Products, and Botanical Products by Liquid Chromatography-Tandem Mass Spectrometry

Renata Raina-Fulton, Asal Behdarvandan, Aisha A. Mohamad, Anusha Pratheepan, Iman Salehi

University of Regina, Department of Chemistry & Biochemistry, 3737 Wascana Parkway; Regina, SK S4S 0A2, CANADA; renata.raina@uregina.ca

In the last several years we have developed targeted chemical class methods for insecticides and fungicides using gas chromatography-negative chemical ionization-mass spectrometry or liquid chromatography-electrospray ionization-tandem mass spectrometry particularly for extracts obtained from solid sample matrices including those used in atmospheric sampling of pesticides. We selected insecticides (organophosphorus pesticides and neonicotinoid insecticides) and their degradation products, strobilurin and conazole fungicides and other selected difficult to analyze pesticides as case models for assessing the challenges faced in LC-MS/MS analyses of pesticides in extracts obtained from different solid sample types. Acceptable overall recoveries were assessed as 70-120% and <20% RSD for insecticides and fungicides in extracts obtained from different matrices ranging from atmospheric particles and solid sorbents used in gas phase collection of pesticides, bees, bee products, and botanical products (dry powders) including those with high pigment levels. In the assessment of the matrix effects we compared internal standard calibration with standard addition calibration. The percentage matrix effect was determined for over 50 fungicides and insecticides with the severity of the matrix effect defined as soft (\pm <20%), moderate (\pm 20-50%), and severe ($>$ \pm 50%). We also compared the matrix obtained from standard pressurized extraction with that obtained with in-cell (of pressurized solvent-extraction) clean-up utilizing low cost sorbents such as chitosan for various difficult sample matrices. The benefits of subsequent clean-up of extracts with solid-phase extraction will also be illustrated. Calibration range for most pesticides was typically from limit of quantitation (1-10 ng/mL) to 100 ng/mL.

O-14 Improved High Throughput Suspect Screening Analysis of Pesticides using a LC/Q-TOF and Novel Software Tool

Karen E. Yannell and Kai Chen

Agilent Technologies, 5301 Stevens Creek Blvd, Santa Clara, CA 95051 USA; karen.yannell@agilent.com

Traditionally, analyzing a large number of pesticides requires costly calibration using a triple quadrupole mass spectrometer or time-consuming analysis of non-targeted data from high resolution instruments. Neither solution is ideal to detect and quantify pesticides in an efficient, high-throughput, and cost-effective manner. To solve this, a workflow was developed that simultaneously quantifies preferred target pesticides and screens for suspect analytes using a high-resolution quadrupole time-of-flight (Q-TOF) ($R = 30,000$ for m/z 118) and a LC/Q-TOF Screener Tool for fast review of hundreds of pesticides. Using QuEChERS processed broccoli, avocado, strawberry, and black tea, 195 pesticides were added to create five calibrators (5-100 ppb). These analytes were preferred targets with quantitative results while another 182 analytes did not have standards, were not quantified, and considered suspects. The samples were analyzed using reverse phase chromatography and a high-resolution Q-TOF operating in positive ionization mode with MS/MS acquisition. Results show good reproducibility, high signal to noise at the lower level of quantitation, and good linearity for target analytes. Quality control samples containing different levels of target and suspect analytes were analyzed and the spiked compounds were identified with high confidence in an easy manner with the LC/Q-TOF Screener Tool. Unknown strawberry samples ($n=16$) from different regions on the United States were also tested, with pesticides found from trace levels to 300 ppb. Many found pesticides were targets but a few were suspects. Conventional and organic produce were tested and the number of found pesticides followed use trends.

O-15 The Renaissance of Supercritical Fluid Chromatography in Food Pesticide Residue Analysis?

Amadeo R. Fernández-Alba and Victor Cutillas

European Union Reference Laboratory for Pesticide Residues in Fruit and Vegetables; Agrifood Campus of International Excellence (CeIA3), Department of Chemistry and Physics, University of Almería, 04120 Almería, Spain; amadeo@ual.es;

New robust supercritical fluid chromatography coupled to electrospray mass spectrometry (SFC-EI-MS) is proving itself a good alternative to the well-known reverse-phase liquid chromatography. This is because of the important advantages that can be observed in the analysis of pesticide residues in fruit and vegetables; nonetheless, certain limitations have also come to light. The use of CO₂ in the mobile phase and the high flow rates applied provide short run times with the early elution of compounds. The CO₂ returns to its gas state before the flow reaches the ion source; therefore, only a small amount of solvent is consumed. One can also observe that the absence of water greatly reduces the typical ion suppression effect due to improved ion sampling efficiency in the ESI as the flow at the ion source is reduced. From the routine analysis standpoint, the possibility of using "regular" reverse-phase columns is a significant advantage over microflow chromatography. Although some drawbacks can be detected, they do not greatly limit the analytical applications. In this work, we illustrate clear examples of the benefits obtained in LOQs as well as the low matrix effects when analyzing a wide variety of commodities. Finally, an example of the method evaluation is presented along with the analysis of real samples using the proposed method.

O-16 New Efficient Approach for the NL-Acetone Extraction Method for Pesticide Residue Analysis by LC- and GC-MS/MS

Jonatan V. Dias¹, Ionara R. Pizzutti¹, André de Kok, Jos Scholten², Barbara Kiedrowska², Carmem D. Cardoso¹

¹**Center of Research and Analysis of Residues and Contaminants (CEPARC)**, Federal University of Santa Maria (UFSM), Chemistry Department, Santa Maria, RS, Brazil; ionara.pizzutti@ceparc.com.br

²Netherlands Food and Consumer Product Safety Authority - NVWA, Laboratory of Food and Feed Safety, Chemistry Laboratory, R&D Group, National Reference Laboratory (NRL) for Pesticide Residues in Food and Feed, Wageningen, The Netherlands

In this study, an efficient modification of the New Dutch mini-Luke method (NL-method) for the determination of pesticide residues in fruits and vegetables by gas and liquid chromatography coupled to tandem mass spectrometry is presented. The NL-method published recently in 2016 [1], was taken as the basis. We propose a direct injection of the initial acetone extract into the UPLC-MS/MS system, thereby expanding the scope of the method to cover more polar pesticides. We also propose a change in the solvents for the partitioning step, in order to eliminate the use of dichloromethane for phase separation, and also the evaporation step. Also, there is no need for salt addition anymore, which results in a cleaner GC-extract. This improvement avoids the use of chlorinated solvents and allows direct injection of the final, more nonpolar and cleaner organic extract into the GC-MS/MS system, without any cleanup. Initial optimization experiments were performed with 30 selected pesticides, representative of different classes, from which 20 were evaluated by UPLC-MS/MS and 10 by GC-MS/MS triple quadrupole mass spectrometry. Unlike in the NL-method, in this new approach the LC amenable pesticides are analyzed immediately after the initial acetone extraction step. An aliquot of the acetone extract (0.5 mL) was taken, diluted with 0.5 mL of methanol and directly injected into the UPLC-MS/MS. For GC amenable pesticides, the procedure follows the same sequence as the NL-method, but exchanging the petroleum ether and dichloromethane for the same volume of isooctane and toluene, respectively. An aliquot of the organic phase extract was taken and injected directly into the GC-MS/MS, without any evaporation step. The efficiency of this improved approach was demonstrated by method validation for 186 LC- and 164 GC-MS/MS amenable pesticides, respectively, with a wide polarity range. Validation was carried out evaluating linearity, linear range, detection and quantification limits, precision and accuracy (from recovery studies). From the pesticides evaluated by UPLC-MS/MS, 77% (144) showed a recovery within the acceptable range of 70-120% and relative standard deviation lower than 20%, at the lowest spike level of 10 µg kg⁻¹, which is the method quantification limit for these pesticides. For ten (for example boscalid and dimethomorph) and two (cyphenothrin and pyrimethanil) pesticides, the LOQ_m was established at 20 and 50 µg kg⁻¹, respectively. For approximately 90% of the studied pesticides, the matrix effect was within the acceptable range of ± 20%. Results for the pesticides evaluated via GC-MS/MS will also be presented.

O-17 Challenges in the Analysis of Perfluoroalkyl Substances in Food Matrices by LC-MS/MS at sub-ppb Concentration

Bjorn J.A. Berendsen, Erik de Lange, and Stefan P.J. van Leeuwen

Wageningen Food Safety Research, Akkermaalsbos 2, 6708 WB, Wageningen, The Netherlands

The European Food Safety Authority has recently published a new opinion (i.e. risk assessment) in which a lower tolerable weekly intake for the perfluoroalkyl substances (PFASs) perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS) is stated. This has a severe impact on the required performance of analytical methodologies to detect, confirm the identity and quantify these compounds in edible matrices. For example, on basis of this opinion, for PFOA in milk a level of quantification (LOQ) of 3 pg/g is required for effective exposure analysis, which is a factor of 80 lower than before.

Besides the lowering of the required LOQ of the analytical methods, the scope of PFASs that are relevant are expanding. For instance, as a result of the ban of PFOA by the United Nations alternative PFASs are of interest. In The Netherlands, the use of heptafluoropropylene oxide dimer acid (HFPO-DA), also called GenX, is used as an alternative and the trimer acid (HFPO-TA) is suspect of occurring.

The current challenges in targeted analytical approaches for PFAS analysis will be discussed. These include the clean-up of food products and the concentration of extracts to achieve the low detection limits, chemical background that occurs especially for PFOA and finally, severe in source fragmentation that occurs for some alternative PFAS and compromises detectability.

O-18 Identification and Determination of BFRs in Food Contact articles and Food

R. Paseiro-Cerrato^{1,2}, L. DeJager¹, T. H. Begley¹

¹US FDA, Center for Food Safety and Applied Nutrition, 5001 Campus Drive, College Park, MD, 20740, USA

²Joint Institute for Food Safety and Applied Nutrition (JIFSAN), University of Maryland, College Park, MD, 20742, USA

Food contact articles (FCA) must be approved by the FDA before being placed into the US market. They can be approved through the code of federal regulations or using the food contact notification system. In addition, FCA producers should

also follow good manufacturing practices. Several scientific articles related with contaminated FCA with brominated flame retardants (BFRs) in the European Union were published in the past years. These authors suggested that this contamination could come from recycled polymers containing waste electronic and electrical equipment (WEEE). When placing a FCA in contact with food a process of mass transfer occurs. This mass transfer process is commonly known as "migration process" and it is ruled by Fick's second law. Any molecule present in the polymeric matrix of a FCA can potentially migrate into food and may represent a food safety issue. In this experiment, we wanted to investigate if FCA contaminated with BFRs, could transfer BFRs into food and food simulants. For this purpose, analytical methods to identify and analyze BFRs in FCA as well as food and food simulants were developed and validated. The methods for BFRs analysis and the migration process of BFRs from FCA into food and food simulants, will be discussed.

O-19 Quantitative Analysis of Naturally Occurring Compounds Regulated under California's Safe Drinking Water and Toxic Enforcement (Prop 65) Act by SPME-HS

Wiley A. Hall, 4th, SFA of California, 2037 Morgan Drive; Kingsburg, CA 93631, USA; wileyh@safefoodalliance.com

Right-to-know notifications per the Safe Drinking Water and Toxic Enforcement Act of 1986, popularly known as "Prop 65", are quickly becoming ubiquitous and even ignorable in California; but the consequences for companies found to be in violation can be quite serious, including fines of up to \$2500 per violation per day and unfavorable public relations. Of particular concern are a series of compounds: including furan, furfuryl alcohol, and acrylamide, that may not be purposefully added to, nor found, in a raw commodity; but form due to the reaction of heat, sugar and protein. The levels of these compounds can vary greatly within a given commodity depending on environmental conditions and processing method with furfuryl alcohol reported to form at temperatures as low as 50°C. Analysis can be further complicated as there can be many chemically similar compounds (furfural, pentyl-furan, furaldehyde, furanone, and furancarboxaldehyde, are among the furan derivatives that have been identified in dried fruit samples, for example) that can cause false positives if particular care is not taken during analysis. Interconversions between some of these compounds (as well as their polymers) have also been reported. The relative advantages and disadvantages of analyzing furan, furfuryl alcohol, acrylamide and 5-hydroxymethylfurfural by SPME-HS coupled with GC-MS TOF vs solvent extraction and analysis by LC-MS/MS will be presented along with the conditions for sub-ppm quantitation with minimal artefactual effect, as determined by comparing responses isotopically labeled and unlabeled analytes.

O-20 Improved Extraction and Cleanup Prior to HPLC Determination of Glyphosate in Food Samples

Sareeta Nerkar and Maria Ofitserova

Pickering Laboratories, Inc., sareetan@pickeringlabs.com; 1280 Space Park Way, Mountain View, CA, 94043; USA

Glyphosate, the active ingredient in Roundup and the most widely used herbicide in the world. Understanding the scope of glyphosate contamination in our food supply is critical to protecting public health, as more scientific evidence continues to link glyphosate with cancer. In the United States, the Environmental Protection Agency (EPA) regulates Glyphosate and sets the maximum amount of herbicide allowed to be present in assorted crops as well as drinking water. Recent research, however, has raised concerns about Glyphosate safety and its prevalence in the environment. In response to the evidence of increased human exposure to this herbicide, the Food and Drug Administration (FDA) announced it will begin testing in foods including soybeans, corn, milk, and eggs. The AOAC Official Method 2000.05 for Analysis of Glyphosate in Crops describes an easy clean-up procedure using cation-exchange cartridges that was successfully combined with Pickering Laboratories post-column derivatization for analysis in crops such as soy, corn, alfalfa, and sunflower seeds as well as vegetables such as tomatoes and broccoli. The downside of this sample preparation technique is the fairly high volume of the water-based solution used to elute Glyphosate from the clean-up cartridge and consequently long evaporation times. The presented post-column derivatization method for Glyphosate analysis utilizes an accelerated sample preparation to quickly and efficiently analyze Glyphosate in a wide range of foods. Since there were more concerns recently about Glyphosate, we tested Glyphosate in hemp, almond milk, orange juice, rice, honey, wine and soy sauce which shows that this method is sensitive and selective, and it can be easily implemented in any laboratory. The sample prep is so simple and does not need any expensive equipment. The method is robust and shows that it is suitable for analysis of Glyphosate on sub-ppm levels in many types of foods. Accelerated sample preparation improves the throughput of samples and reduces the cost of testing for busy laboratories. The sensitivity of the method allows for the detection of Glyphosate well below residue tolerances set by regulatory agencies.

O-21 Colorado Department of Agriculture Laboratory Update

Eric Petty, Keith Wegner, Kristina McCallum, and [Jessica Krank](mailto:Jessica.krank@state.co.us)

Colorado Department of Agriculture, Division of Laboratory Services, 300 South Technology Court; Broomfield, CO 80021, USA; Jessica.krank@state.co.us

The Colorado Department of Agriculture has experienced considerable change in the last year. The role of pesticide testing related to cannabis compliance and enforcement, applicator enforcement, ground and surface water monitoring continues to expand. With the passage of the Farm Bill and the designation of Hemp as an agricultural commodity, applications for permits to grow hemp and the required THC testing continues to escalate. In January, 2019 the various laboratories of the Department were consolidated into a new Division of Laboratory Services. In April of this year the Division relocated 30 miles North of Denver and moved into a new 22,000 square foot laboratory facility located on the Colorado Department of Agriculture campus.

O-22 nDATA workflow for Screening Pesticides in Fruits and Vegetables

Jon Wong, **U.S. Food and Drug Administration**, College Park, MD, USA

A non-target data acquisition for target analysis (nDATA) workflow based on accurate mass measurements using UHPLC/ESI Q-Orbitrap full MS-data-independent acquisition (DIA) and a compound database was developed to screen pesticides in fruits and vegetables. A compound database of over 1000 pesticides was built from data-dependent acquisition (DDA) product ion spectral data, accurate mass precursor ions and LC retention times of individual pesticide standards. Full MS-variable DIA (vDIA) or full MS-multiplex DIA (mDIA) methods were used to acquire sample data from ten fruit and vegetable matrices fortified with pesticides and processed by QuEChERS sample preparation. Screening of pesticides in samples was achieved based on the Retention Time (± 0.5 min) and the mass accuracy (± 5 ppm) of the Precursor ion (RTP by full MS) or the Retention Time (± 0.5 min) and the mass accuracy of the precursor and corresponding Fragment Ion(s) (± 10 ppm) (RTFI by full MS/DIA). Of the 845 pesticides studied, RTP correctly detected up to 765 and 796 pesticides at 10 and 100 $\mu\text{g}/\text{kg}$, respectively and 729 and 764 pesticides by RTFI at 10 and 100 $\mu\text{g}/\text{kg}$, respectively. To test the transferability of the nDATA workflow, a collaborative study evaluating ten variably fortified produce samples using a 50-pesticide compound database was coordinated. Preliminary results demonstrate the participating laboratories, including six state laboratories (California, Connecticut, Indiana, Minnesota, North Carolina and New York Department of Health), that submitted their results were able to successfully complete the qualification, validation, and proficiency phases of the study.

O-23 A High Resolution Mass Spectrometry (HRMS) Method for More 1000 Pesticides and other Poisons: The Method and Madness

Marc Engel, **FDACS**, Tallahassee, FL, USA

Approximately 6 billion pounds of pesticides are used annually worldwide and about 1 billion pounds are used annually in the USA (USEPA). To keep food supply and the environment safe the modern regulatory laboratory must be able to detect and uniquely identify contaminants. High resolution mass spectrometry (HRMS) plays a key role in the identification and quantification of potential contaminants, since it is impossible for any laboratory to have large numbers individual standards. This work describes an ongoing project to develop a HRMS pesticide/poison screen using HRMS to screen 1000 or more pesticides and other toxic substances. Sixteen pesticide mixes were analyzed by LC HRMS using a Exactive Plus Orbitrap. A 32-minute water with 0.1% formic acid and acetonitrile with 0.1% formic acid LC gradient is being used. The HCD is run at 35k resolution at 15eV and 45 eV. Four-hundred pesticides were added to a preexisting screen from these mixes by extracting the "exact mass" of the precursor ion and aligning strong responding product ions in a calculated retention time window.

In addition, the mixes were analyzed with the same buffers, but a 62 minute gradient using a Q-Exactive at 35k resolution in full scan and with a series of PRM experiments. The CE stepped at 15eV, 30 eV and 45 eV for the PRM experiments.

O-24 Analysis of Sodium Fluoroacetate (1080) and Dicamba using the Gerstel Multi Purpose Sampler DHS and Twister® capabilities

Jean-Paul Schirlé-Keller and Yoko Johnson

Minnesota Department of Agriculture, Environmental analysis laboratory, 601 N Robert Street, Saint Paul, MN, 55155; USA

Sodium Fluoroacetate (NaFAC) is a poison with LD50 of 0.1 ppm and Dicamba is currently an herbicide generating high numbers of samples to analyze. The current available methodology for NaFAC has LOD of 10 ppm by SPME. Dynamic headspace was used to lower these LODs in beverage and food systems. Volatiles were trapped on Tenax 60/80 sorbent using a Gerstel Dynamic Headspace System and subsequently analyzed by GC-MS. LOD NaFAC was decreased to 20ppb in water when using the original protocol. These levels varied when measured in milk, apple juice, beer, hotdog, cooked pork, raw chicken and raw beef respectively. Dicamba current LOD is 0.5ppb on-column using LC-HRMS. However, its extraction protocol is lengthy. Twisters® technology was investigated as screening technique for Dicamba analysis. When directly immersed in pH = 1 Dicamba standard solutions, Twisters exhibited 1ppb LOD by GCMS analysis. This technology successfully isolated Dicamba when twisters were immersed in soil solutions for which the MRL is 100ppb. However, it failed to reveal the presence of Dicamba in vegetation extracts due to strong interactions of plant organics with the twisters.

O-25 Multiresidue Method for Analysis of Veterinary Drug Residue In Meat By LC-HRMS - Screening and/or Quantitation?

Dominique Hurtaud-Pessel, Pierrick Couedor, Eric Verdon

Anses, French Agency for Food, Environmental and Occupational Health And Safety, 35300 Fougères, France; dominique.pessel@anses.fr

Liquid chromatography coupled to high-resolution mass spectrometry is a valuable tool for the multi analyte determination of residues since it allows detecting an almost unlimited number of different compounds during the same analytical run. This asset is therefore particularly well suited to screening methods. As part of our Reference laboratory activities for regulatory control of veterinary drug residues in food from animal origin, a method was developed for the screening in meat of 145 compounds belonging to 5 main classes of veterinary drugs: antibacterials, anthelmintics, NSAIDs, sedatives and coccidiostats. Full scan combined with fragmentation mode using variable data-independent acquisition (v-DIA) was operated to get identification based on accurate mass of parent and fragment ions. A single validation process was applied to assess the performances of the method for both screening and quantitative purposes. The results showed that the method allows detecting about 80% of the compounds at the lowest spiking level of 10 µg/kg. The potential of the method for quantitative analysis has been evaluated through measurement trueness and precision over three spiking levels. The added advantage of HRMS with the ability to retrospectively analyze data has also been tested through participation in a European proficiency test and showed that the method can be easily extended to a wider range of compounds, and also to be applied to milk.

O-26 Development and Validation of a QuEChERS Mega-Method for the Analysis of Pesticides, Veterinary Drugs, and Environmental Contaminants in Fish and Meat

Steven J. Lehotay,¹ Sergio Monteiro,² Ederina Ninga,³ and Yelena Sapozhnikova¹

¹ USDA Agricultural Research Service, 600 East Mermaid Lane; Wyndmoor, PA 19038, USA; steven.lehotay@usda.gov

² Biological Institute, Environmental Protection Research Center; Sao Paulo; Brazil

³ Food Safety and Veterinary Institute, Dept. of Toxicology and Residues Monitoring; Tirana; Albania

The most efficiency is achieved in laboratories by combining more analytes into a single method, including analytes from different applications for the same matrix. A large number of veterinary drugs, pesticides, and environmental contaminants often need to be monitored in animal-derived foods, such as fish, meats, milk, eggs, and honey.

Conventionally, separate methods have been used for each type of application, but modern techniques and instruments can accommodate simultaneous analysis of >300 analytes at >5 ng/g levels in the complex matrices. We merged our two separate QuEChERS-based methods for veterinary drugs and pesticides into a single method covering >350 pesticides, veterinary drugs, and environmental contaminants, including main metabolites, in fish and meats. In the method, 2 g comminuted sample is extracted with 10 mL of 4/1 (v/v) acetonitrile/water for 10 min by shaking, followed by centrifugation. A small portion of the initial extract is diluted in water for analysis of veterinary drugs and polar pesticides by UHPLC-MS/MS, and the remaining extract is decanted into a 15 mL tube containing 1 g of 4/1 (w/w) MgSO₄/NaCl, which is shaken 1 min and centrifuged again for 3 min. For analysis of GC-amenable pesticides and environmental contaminants, 1 mL of the upper layer is transferred into an autosampler vial, 300 µL of which undergoes cleanup by micro-solid-phase extraction (µ-SPE) using automated Instrument Top Sample Preparation (ITSP), immediately followed by low pressure (LP) GC-MS/MS analysis. ITSP is conducted in parallel with LPGC-MS/MS, which is also conducted in parallel with UHPLC-MS/MS, with all methods taking <15 min cycle times per sample. For MS/MS on both instruments, 3 ion transitions are monitored to improve identification of the targeted analytes, and many pesticides are analyzed on both instruments, providing additional confidence in results using orthogonal information. The new QuEChERS mega-method can be used for a narrow or wide scope of analytes and matrices, depending on the application need, rather than using a different method for each application.

O-27 Occurrence of Residues of Veterinary Antibiotics in Water, Sediment and Trout Tissue (*Oncorhynchus Mykiss*) in the South Area of Titicaca Lake – PERU

Franz Zirena Vilca^{1 2 5} Nestor Cahui Galarza², Juan Rigoberto Tejedo Huaman³, Walter Alejandro Zamalloa Cuba^{2 4}, Clara Nely Campos Quiroz⁵ and Valdemar Luiz Tornisielo⁶

¹ Escuela Profesional de Ingeniería Ambiental de la Universidad Nacional de Moquegua, Urb Ciudad Jardín-Pacocha-Ilo-Perú; franzvilca@usp.br

² Escuela de Posgrado de la Universidad Nacional del Altiplano Puno Av. Floral 1153 Puno-Perú.

³ Regeneration and Cell Therapy Department Andalusian Center for Molecular Biology and Regenerative Medicine – CABIMER. Parque Científico Tecnológico Cartuja Edificio CABIMER Avda Américo Vespucio N° 24 41092 Seville, Spain.

⁴ Escuela Profesional de Ingeniería Química de la Universidad Nacional del Altiplano, Av Floral S/N Puno-Perú

⁵ Universidad Nacional Toribio Rodríguez de Mendoza de Amazonas, Calle Higos Urco N° 342-350-356, Calle Universitaria N° 304, Chachapoyas – Amazonas-Perú

⁶ Centro de Energía Nuclear na Agricultura, Laboratorio de Ecotoxicología, Avenida Centenario 303, 13416-903 Piracicaba, SP, Brasil

The production of trout (*Oncorhynchus mykiss*) in Peru has experienced significant growth in the last decade, being the city of Puno the largest producer through intensive systems in floating cages installed in Lake Titicaca. As a consequence it has been described the increase of diseases and the use of antibiotics to control them during the production cycle. In this work we study the impact of antibiotics on drinking water, trout tissues and the lake's aquatic ecosystem. A total of nine veterinary antibiotics were monitored: tetracyclines (chlortetracycline, oxytetracycline and tetracycline), sulfonamides (sulfathiazole, sulfamethazine and sulfadimethoxine) and fluoroquinolones (ciprofloxacin, enrofloxacin, and sarafloxacin). The samples were collected using a non-probabilistic system and analyzed by liquid chromatography coupled to mass spectrometry and solid phase extraction on line (On-line SPE-LC-MS/MS). The sediment samples showed residues of fluoroquinolones (3739.3 µg kg⁻¹) and tetracyclines (3082.9 µg kg⁻¹). Similarly, surface water samples showed concentrations of fluoroquinolones of up to 408.2 and 652.7 ng L⁻¹ in dry and rainy seasons respectively (P>0.05) and samples of drinking water reached an average of 188.1 and 222.2 ng L⁻¹ of ciprofloxacin in dry and rainy seasons respectively. While in trout tissues reached 7.8 µg kg⁻¹ in oxytetracycline 8.7 µg kg⁻¹ in sulfatizole, 4.2 µg kg⁻¹ in ciprofloxacin and 3.6 µg kg⁻¹ in sarafloxacin (P>0.05). The presence of these antibiotics in the trout tissue is attributed to the constant use of these drugs for the prevention and treatment of diseases within the productive system; their presence can have detrimental effects on the aquatic ecosystem, and can even affect public health due to the consumption of aquaculture products and drinking water contaminated with antibiotic residues.

O-28 Occurrence of Antibiotics and Veterinary Drug Residues in Wildlife

Ovokeroye A. Abafe

Department of Chemical Residue Analysis, Agricultural Research Council-OVR, Private Bag X05, Onderstepoort, Pretoria 0110, AbafeO@arc.agric.za

Antibiotics are one of the biomedical revolutions of the 20th century. However, their misuse has led to an increase in diseases in humans, domestic animals and wildlife worldwide. Huge quantities of antibiotics are used annually in livestock farming operations throughout the world, but the ultimate fate of their residues and their potential damage to environmental and human health generally remains unknown. In this study, the presence of antibiotics and veterinary drug residues in wildlife – Crocodile, Ostrich and Game were measured using a validated method based on Liquid Chromatography tandem Mass spectrometry (LC-MS/MS).

The levels of antibiotics ranged from 21.67 – 108.60 µg/kg Oxytetracycline in Crocodile tissues; 713 µg/kg tetracycline, 791 µg/kg Chlortetracycline, 730 µg/kg Oxytetracycline and 789 µg/kg Doxycycline in the tissue of one Ostrich. Lincomycin (18.83 µg/kg) was found in one Ostrich and Abamectin (121 µg/kg) and Ivermectin (78.63 µg/kg) was reported in the tissue of one Crocodile sample. None of the antibiotics and veterinary drug was found at measurable levels in any of the eighty three Game tissue samples.

Though limited literature are available on the occurrence of antibiotics and veterinary drugs in wildlife, the presence of residues of these antibiotics in wildlife may contribute to the incidence of antibiotics resistance in wildlife.

O-29 The Need for Safety and Authenticity Testing Methods for Natural Colors

Joe Konschnik¹, Mark Goldschmidt², Doug Williams³

¹Business Development Manager, **RESTEK Corporation**, Bellefonte, PA, USA; joe.konschnik@restek.com

²Global Quality Control & Product Safety Director, Sensient Technologies, Milwaukee, WI, USA; Mark.Goldschmidt@sensient.com

³Research Fellow, Kalsec, Inc., Kalamzoo, MI, USA; DWilliam@kalsec.com

Color additives are applied in a broad range of products that impact our lives. With up to 85% of consumers buying decisions potentially influenced by color, appropriate application of color is critical to product selection and experience. From a quality and safety perspective, for colors from natural sources there is a general lack of product definitions or publicly available purity, quality and safety specifications that are consistently applied. Under current regulations, color additives fall into two categories: Those subject to the FDA's certification process, and those that are exempt. Recently, a growing consumer interest in "natural colors" has led to a broad application of plant extracts and other materials as "exempt" colorants in foods and beverages. A significant risk of adulteration of natural colors exists, ranging from simple misbranding or misuse of the term "natural" on a product label to potentially serious cases of physical, chemical, and/or microbial contamination from raw material sources, improper processing methods, or intentional postproduction adulteration. Consistent industry-wide safety standards are needed to address the manufacturing, processing, application, and international trade of colors from natural sources to ensure quality and safety throughout the supply chain. Methods are needed for the analysis of residual solvents, microorganisms, heavy metals, pesticides, and mycotoxins as a means to establish such standards. The focus of this presentation is to highlight the expanding use of natural colors in the food industry and discuss key potential safety hazards affecting sourcing and use of food colorants from natural sources.

O-30 Perfluoroalkyl compounds in Milk and Maple Syrup

Robert Sheridan, **New York State Department Of Agriculture**, Albany, NY, USA

Perfluorinated compounds (PFCs) have been produced since the 1940's and used in many industrial and commercial applications due to their unique properties. They have been used for decades as water and oil repellants, insulators in electrical wires and in firefighting foam. In 2000 the manufacturer of these compounds began the phaseout of the two most commonly used PFCs Perfluorooctanoic acid (PFOA) and Perfluorooctanesulfonic acid (PFOS) due to safety

concerns however because these compounds are extremely stable they persist in the environment where they can pass through the food chain. In geographical areas of PFOA and PFOS groundwater contamination there is concern that these compounds make their way into locally produced food. Milk and maple syrup are produced in upstate New York and require a large amount of water which could provide a possible source of contamination. A method for the measurement of PFOA and PFOS in milk and maple syrup was developed and applied to the analysis of sample collected in 2017. The milk extraction method employs liquid liquid separation followed by dispersive solid phase clean-up while the maple syrup extraction method uses SPE. PFOA and PFOS are quantified using UPLC triple quadrupole MS.

O-31 Advances and Data Gaps in Analytical Discovery, Detection, and Quantitation of Algal Toxins

Keith A. Loftin, Zachary R. Laughrey, Ariel R. Donovan

U.S. Geological Survey, Kansas Water Science Center, Organic Geochemistry Research Laboratory, 1217 Biltmore Dr., Lawrence, KS 66049

Harmful algal blooms and the toxins they sometimes produce are perceived to be increasing in prevalence and severity across the globe. Illness after toxin exposure in animals and humans has been documented. In more severe cases of exposure, mortality has been observed in wildlife, livestock, and companion animals with a few notable cases of human death attributed to toxin exposure outside of the United States. A variety of algal toxin chemical classes with different modes of action exist. These include cyanotoxins such as anatoxins, cylindrospermopsins, microcystins, nodularins, and saxitoxins produced by cyanobacteria and marine algal toxins typically produced by diatoms and dinoflagellates such as azaspiracids, brevetoxins, dinophysistoxins, domoic acids, gymnodimines, pectenotoxins, okadaic acids, saxitoxins, and spirolides. While assays such as enzyme-linked immunosorbent assay are commonly used for screening, liquid chromatography-mass spectrometry is one of the more common approaches to obtain compound specificity, robust quantitation, and unknown toxin discovery. Algal toxins vary in physical properties and are not usually well characterized due to the difficulty and expense in obtaining pure standards in sufficient quantity. Many of these physical properties, including metal chelation, hydrophobicity, as well as the formation of multiple charge states, can adversely impact minimum reporting levels of algal toxins. Specific advances and data gaps in analytical methods development and validation will be discussed, including sample preparation, matrix effects, and adduct formation that can adversely impact minimum reporting level.

O-32 Emerging Algal Toxins in California: Responding to Known Threats, Preparing for the Future

Raphael M. Kudela,¹ Melissa Peacock²

¹**University of California Santa Cruz, Ocean Sciences Department**, 1156 High Street; Santa Cruz, CA 95064, USA; kudela@ucsc.edu;

²Northwest Indian College; Bellingham, WA USA;

Harmful Algal Blooms (HABs) have emerged as an increasing threat to California ecosystem health, with frequent and widespread impacts to both wildlife and humans. We are increasingly recognizing that the west coast is not immune to numerous unrecognized HAB issues. For example, direct transfer of freshwater toxins have been associated with deaths of California sea otters and dangerously high levels of toxins in recreationally and commercially harvested shellfish. Recent surveys of wide swaths of California have highlighted that these are not isolated incidences, but severely under-reported threats driven by interactions at the land-sea interface where freshwater and marine toxins mix. A fundamental requirement for monitoring and management of these issues is to develop a more holistic approach to understanding and predicting these events, i.e. not treating each HAB as an ecologically unique issue. We propose that Solid Phase Adsorption Toxin Tracking (SPATT) could be leveraged to effectively address the science/management gap that exists regarding the simultaneous occurrence of multiple toxins. SPATT that are already routinely collected as part of existing projects could be used to derive much more information about the biological community, environmental drivers, and patterns leading to HAB events by statistical modeling of multiple toxins from the same SPATT in conjunction with other environmental data. This requires adaptation of existing LCMS methods, and perhaps adoption of a modified SPATT design suitable for a wide range of toxins. Here we present preliminary results and recommended methods for use of SPATT in trace analysis of environmental algal toxins.

POSTER ABSTRACTS

P-3 Determination of Drugs of Abuse in Human Hair by On-line Supercritical Fluid Extraction – Supercritical Fluid Chromatography - Mass Spectrometry

A. Paige Wicker,¹ Blair K. Berger,¹ Tairo Ogura,² Kenichiro Tanaka,² Masayuki Nishimura,³ Vivian Chen,³ William Hedgpeeth,³ and Kevin A. Schug;¹

¹ The University of Texas at Arlington; Department of Chemistry & Biochemistry, Arlington, TX 76010, USA; blair.berger@mavs.uta.edu

² Shimadzu Corporation, Nishinokyo Kuwabara-cho, Nakagyo-ku, Kyoto 604-8511, Japan.

³ Shimadzu Scientific Instruments, Inc, Innovation Center, 7102 Riverwood Drive, Columbia, MD 21046, USA.

The detection of drugs of abuse (DoA) in hair; being a convenient and noninvasive technique for determination of controlled substances, is important in forensics and toxicology. On-line supercritical fluid extraction – supercritical chromatography (SFE-SFC) coupled with mass spectrometry (MS/MS) is a quickly developing technique for the extraction, separation, detection, and quantification of analytes in a single analysis. SFE-SFC-MS/MS ideal for DoA determination in complex matrices, provides highly specific and sensitive chemical analysis while limiting the need for extensive manual sample preparation. Supercritical fluid extraction of 12 DoAs from hair was performed on-line using carbon dioxide (CO₂) with 5 mM ammonium formate in methanol for chromatographic separation on HILIC-Si column [150 mm x 4.6 mm, 2.7 μm (Restek Corp.)] at 50 °C. Dual, variable, back pressure regulators (BPRs), pre- and post-column, were used to ‘trap’ extractant at the column head. Multivariate analysis, was used to optimize extraction parameters, including time (static and dynamic), modifier concentration, flow rate and pressure. It was determined that a compromise between flow rate and concentration was needed. Optimal extraction occurred using 30% modifier at 5.00 mL/min for 8 minute static and 15 minute dynamic flow at 30 °C with dual backpressures of 10.0 MPa. Backpressures were increased to 40.0 MPa (pre-column) and 15.0 MPa (post-column) directing flow to the column. Five point calibration curves (n=3) were created in the range of 10–400 pg/mg DoA in human hair. Linearity of R² ≥ 0.99 achieved. Method validation is currently underway to ensure detailed quantitative analysis for hair samples.

P-4 Persistent Organic Pollutants in Lakes of Grovnes Peninsula at Larsemann Hill Area, East Antarctica

Laxmikant Bhardwaj and Professor Tanu Jindal

Amity Centre for Antarctic Research and Studies and Amity Institute of Environmental Toxicology, Safety and Management, Sector-125, Amity University, Noida, Uttar Pradesh, INDIA

Over the past decades, research in Antarctica has built a new understanding of its past, present, and future. Human activities are increasing on Antarctica because of various scientific expeditions. Research on Persistent Organic Pollutants (POPs) has been carried out internationally by several countries having their permanent research station to explain the impact of an ever increasing range of POPs in the Antarctic ecosystem. Additionally, global pollution due to various newly introduced pollutants like pesticides is on use since the past century and many factors contribute to contamination even in Antarctica.

More than 150 lakes at different island and peninsulas are situated in Larsemann Hill, East Antarctica. It is a series of islands and rocky peninsulas which consists of two major peninsulas, four minor peninsulas and ~ 130 near-shore islands. POPs are semi-volatile toxic compounds which resist from photolytic, chemical and biological degradation, can persist in the environment for a long time. POPs were analyzed in the Lakes water samples of Grovnes peninsula, Larsemann Hills during 34th Indian Scientific Expedition to Antarctica (ISEA) in austral summer of 2014 to 2015. POP's residue levels were found in lake water samples varied from 10.00 to 75.00 pg/mL. Presence of p,p'-DDT was detected in all different lakes & highest concentration was found in L1E NG lake. The presence of POPs may be attributed to orographic effects, migratory birds, biomagnification and anthropogenic sources. The presence of POPs is an alarming situation and needs to be investigated further to maintain the pristine environment in Antarctica.

P-5 Evaluation of Sample Preparation Techniques for Cannabis and Cannabis Products

Kelsey Cagle and Frank L. Dorman

Department of Biochemistry Microbiology and Molecular Biology, 103 Althouse Laboratory, Penn State University, University Park, PA 16802

This poster presentation will cover various sample preparation techniques for cannabis and cannabis products and contain an evaluation of their overall effectiveness in pesticide recovery. This study aims to provide a basic overview of sample preparation techniques so that the appropriate method can be chosen in future work depending on what the goals are of that work.

Each matrix presents a particular challenge associated with it and those challenges will in turn determine the possible sample preparation techniques that would provide viable results. Ultimately, finding a preparation technique that can effectively minimize or solve the problems with a particular matrix is the goal for any method development. The main difficulty found with botanical matrices is the homogenization of the sample itself. The samples will undergo 3 separate extraction methods, all 3 variations using a SampliQ C18 SPE cartridge after homogenization. Edibles produce an interesting challenge, as many of these matrices will have a high fat content that could interfere with the analysis. The main goal of the extraction process for edibles is to remove or diminish the amount of lipid and lipid-like compounds present in the sample, which can be accomplished using various EMR-Lipid methods. The pesticides in edibles will be extracted using a Bond Elut EMR-Lipid method as well as a separate Captiva EMR-Lipid method. Cannabis candy products are also challenging to work with as dissolving the sample in order to extract the pesticides can often be time consuming and problematic. The candy matrix will have the pesticides extracted using a QuEChERS method followed by one of these subsequent methods- Bond Elut PSA filtration, SampliQ C18 SPE cartridge filtration, or a HF BondElut C18 SPE cartridge filtration.

In order to support the conclusions being made in this presentation regarding the most effective sample preparation techniques, both GC-MS/MS and LC data will be collected for each method. The resolution, sensitivity, and recovery of pesticide residues and cannabinoid potency associated with each sample preparation method will be evaluated.

P-6 Exploring the efficiency of various extraction approaches for determination of crude (4-methylcyclohexyl) methanol (MCHM) constituents in environmental samples

Ronald V. Emmons,¹ Amila M. Devasurendra,^{1,2} Nipunika H. Godage,¹ and Emanuela Gionfriddo;^{1,3}

¹Department of Chemistry and Biochemistry, University of Toledo, 2801 W. Bancroft; Toledo, OH 43606, USA; emanuela.gionfriddo@utoledo.edu;

²School of Public Health, University of Michigan; Ann Arbor, MI, USA;

³School of Green Chemistry and Engineering, University of Toledo; Toledo, OH, USA

Crude (4-methylcyclohexyl)methanol (MCHM) is a chemical blend mainly used in the coal industry for the separation of usable coal from rocks, debris and coal dust by froth flotation. Crude MCHM consists of (4-methylcyclohexyl) methanol, 4-(methoxymethyl)cyclohexanemethanol, methyl 4-methylcyclohexanecarboxylate, dimethyl-1,4-cyclohexanedicarboxylate, and 1,4-cyclohexanedimethanol.

In 2014, a spill of crude (4-methylcyclohexyl)methanol (MCHM) into Elk River resulted in the contamination of the drinking water of 300,000 residents in West Virginia and Kentucky. In response to this spill, various studies demonstrated that sorbed MCHM readily desorbed from polyethylene into water at levels above the odor threshold, confirming the risk of its long-term exposure to residents from contaminated tap water pipelines. Considering this, it is imperative the development of analytical methods able to detect crude MCHM components in environmental water samples to provide an effective analytical tool for water quality monitoring. In this work, two microextractive methods based on SPME in fiber format and TFME were developed and validated. Their performance was compared with a modified SPE method based on EPA Method 522 for analysis of volatiles in water. SPME and TFME methods both showed enhanced performances in terms of achievable LOQs compared to the SPE protocol (SPE 0.25 – 25 mg L⁻¹; SPME 0.25 – 2.5 µg L⁻¹; TFME 0.1 – 2 µg L⁻¹). Moreover, the sensitivity of the TFME method coupled with its higher analytical throughput established TFME as the optimal extraction approach for crude MCHM constituents and a metabolite of 4-MCHM, with limits of quantitation below the odor threshold for aqueous crude MCHM (0.55 µg L⁻¹).

P-7 Silicone based spray adjuvants in agriculture and their characterization by liquid chromatography coupled with high-resolution mass spectrometry

Serhan Mermer,^{1,3} Raymmah A. Garcia,^{2,3} Vaughn M. Walton,¹ Jennifer A. Field,³

¹Department of Horticulture, Oregon State University, 4017 Ag and Life Sci. Bldg. Corvallis, OR, USA, 97331; mermers@oregonstate.edu

²Department of Chemistry, Oregon State University, 153 Gilbert Hall, Corvallis, OR, USA, 97331

³Department of Environmental and Molecular Toxicology, Oregon State University, 1007 Ag and Life Sci. Bldg. Corvallis, OR, USA, 97331

Spray adjuvants have been used in agriculture since 1950s as penetrants, spreaders, and wetting agents to improve the efficiency of pesticide spray applications. Current literature shows that some classes of surfactants (e.g. hydrocarbon and silicone-based surfactants) have been identified in commonly used spray adjuvants in addition to environmental matrices i.e. beeswax, pollen, almond flowers, and surface water. Even though some classes of surfactants are known, the other components are unknown due to the proprietary nature of spray adjuvant formulations.

In this body of work, commonly used spray adjuvants, Kinetic and Silwet L-77 as well as commercially available reference materials (502W) were characterized by liquid chromatography coupled with high-resolution quadrupole time of flight mass spectrometry (LC-qTOF). Kendrick Mass Defect plots in conjunction with enviMass™ were used to tentatively identify the suspect classes of surfactant present in common spray adjuvants.

Preliminary results indicated that substituted trisiloxanes with CH₃, COCH₃ and H functional groups were found including in Kinetic and Silwet L-77 which was consistent with precedent literature. Upcoming work is to focus on the suspect compounds by using additional non-target identification tools to broaden our knowledge.

P-9 An Emerging Technology for Elevating Concentration of Glucosinolates Biofumigant Fungicides

George Antonious, Kentucky State University, Frankfort, KY, USA; george.antonious@kysu.edu

Biofumigation is the integration of plant residues that release isothiocyanates (ITCs) compounds into the soil as they decompose. One of the recent technology of biofumigation is to assess the impact of agricultural soils incorporated with animal manure and recycled waste on concentrations of organic glucosinolates (GSLs) that control soil-borne diseases. Mixing Brassica dry or fresh plant tissues into agricultural soil defeats soil-borne pests and diseases owing to the release of ITCs, the most effective product of GSLs hydrolysis. A simple, inexpensive, and accurate method was developed for extraction, separation and quantification of GSLs in Brassica plants roots and shoots. GSLs in the shoots and roots of were extracted with boiling methanol to inhibit endogenous myrosinase (the enzyme that hydrolyze GSLs). GSLs separated by adsorption on a diethylamino-ethyl ether ion exchange resin were quantified based on measurement of enzymatically released glucose upon hydrolysis of GSLs. Results revealed that plants grown in soil mixed with sewage sludge (SS) contained greater GSLs content (1287 mg g⁻¹ fresh shoots) compared to plants grown in no-mulch (NM) bare soil (981 mg g⁻¹ fresh shoots). The increase in GSLs concentration indicated that leaves of plants grown in soil amended with SS could play a significant role in controlling soil-borne diseases in conventional and organic agriculture to replace the use of synthetic soil fumigants, such as the hazardous metam sodium, methyl bromide, and ethylene dibromide. Biofumigation in horticultural crops has increased due to prevention of some synthetic soil biofumigants in the U.S. and worldwide. This need makes the use of organic GSLs in soil fumigation an emerging technology for organic and conventional agriculture, human health, and environmental quality.

P-10 Development, validation and implementation of a GC-EI-Orbitrap HRMS method for routine analysis of pesticide residues in fruits and vegetables

Barbara Kiedrowska, André de Kok, Sonia Herrera López, Jos Scholten

WFSR – Wageningen Food Safety Research, National Reference Laboratory (NRL) for Pesticide Residues in Food and Feed, Wageningen, The Netherlands; barbara.kiedrowska@wur.nl; andre.dekok@wur.nl

Fast and comprehensive full scan accurate mass high resolution (HRMS) analysis has become available recently as an

alternative for the governmental and private pesticide residue laboratories responsible for providing residue data for enforcement, the EU-coordinated monitoring program or official EU import control program. Nowadays, high resolution instruments provide a unique combination of excellent mass-accuracy and high resolving power, as well as good sensitivity and selectivity, allowing laboratories to analyse the most challenging matrices with a practically unlimited scope. Additionally to the high number of target analytes, the technique takes advantage of optional identification of unknowns and retrospective analysis, which helps research scientists answer today's most challenging analytical questions. However, every technique has some limitations.

The aim of this study was to develop a GC-EI-Orbitrap multi-residue method for the determination of pesticides in fruits and vegetables and more difficult matrices such as tea, spices and herbs. The GC-EI-Orbitrap method is intended to be complementary to the GC-MS/MS (TQ-EI) method and to reduce false-negative and/or false-positive results for the analysis of complex matrices.

The EU SANTE AQC document criteria for HRMS identification will be critically evaluated, based on the full validation data. Routine analysis results of the GC-Orbitrap will be compared with GC-MS/MS Triple Quad results, as to sensitivity (method LOD, LOQ, LOI), selectivity and false-positives/negatives, for the analysis of fruits and vegetables. Typical examples of monitoring and survey results will be shown.

P-11 Quantitative Analysis Glyphosate, Glufosinate-ammonium, and 3-MPPA in Corn, Wheat and Soybeans Using Ultra High Performance Liquid Chromatography / Tandem Mass Spectrometry with Electrospray Ionization

James M. Krest, Tim M. Brewer, Justin Tone-Pah-Hote and Thomas A. Weber

USDA, Agricultural Marketing Service, Federal Grain Inspection Service 10383 N. Ambassador Dr., Kansas City, MO 64153, USA; timothy.m.brewer@usda.gov;

Glyphosate or N-(phosphonomethyl) glycine is one of the most widely used herbicides with applications in agriculture, forestry, industrial weed control, and lawn and garden. Glyphosate is a non-selective systemic herbicide that can be applied directly to plant foliage. The Environmental Protection Agency tolerance for glyphosate for corn, wheat, and soybeans includes both the parent plus N-acetyl-glyphosate, calculated as the stoichiometric equivalent of glyphosate. The Federal Grain Inspection Service (FGIS) has established a robust glyphosate analysis method in corn, wheat, and soybeans based on derivatization with an analytical range from 10 to 2000 ng/g. The method also provides for the analysis of glufosinate ammonium, and 3-methylphosphinopropionic acid (3-MPPA) with Limits of Reporting (LOR) of 50 ng/g for each. The method is used for the analysis of corn, wheat, and soybean lots in support of FGIS's mission to facilitate the marketing of United States grain.

P-12 Pesticides Analysis Services offered by Federal Grain Inspection Service

Tim M. Brewer, James M. Krest, Angela Mitchell, Matthew Rush, Joseph Davidson and Thomas A. Weber

USDA, Agricultural Marketing Service, Federal Grain Inspection Service 10383 N. Ambassador Dr., Kansas City, MO 64153, USA; timothy.m.brewer@usda.gov

The Federal Grain Inspection Service (FGIS) helps move our Nation's harvest into the marketplace by providing farmers, handlers, processors, exporters, and international buyers with sampling inspection, process verification, weighing and stowage examination services that accurately and consistently describe the quality and quantity of the commodities being bought and sold. One way that FGIS ensures the quality of United States grain is through our Pesticides Analysis Services (PAS) program. Plant protection products (more commonly known as pesticides) are widely used in agriculture to increase the yield, improve the quality, and extend the storage life of food crops. Pesticides must undergo extensive efficacy, environmental, and toxicological testing to be registered by governments for legal use in specified applications. The applied chemicals and/or their degradation products may remain as residues in or on the agricultural products, which becomes a concern for consumer exposure. FGIS provides a pesticide residue testing service for grains under the PAS Program which is described in Program Directive 9180.40. Currently, FGIS offers pesticide testing for three commodities (corn, soybeans and wheat) encompassing ten pesticide analysis methods. The service is provided using gas chromatography/mass spectrometry or gas chromatography/tandem mass spectrometry for listed routine compounds and liquid chromatography/tandem mass spectrometry for special compounds. FGIS analyses for 61 pesticides residues in corn, 74 pesticides residues in wheat, and 103 pesticide residues in soybean. Presented here are the pesticide analysis methods that are offered by FGIS in support of FGIS's mission to facilitate the marketing of United States grain.

P-13 Trace concentration determination of phthalates in non-PVC food packaging

Katherine S. Carlos, Lowri de Jager and Timothy Begley

US FDA, Center for Food Safety and Applied Nutrition, 5001 Campus Drive, College Park, MD, 20740, USA; Katherine.Carlos@fda.hhs.gov;

PVC is a common food contact material that can be plasticized to increase its flexibility. Phthalates are one of many chemical compounds that are used as plasticizers in PVC. Transfer of plasticizers from packaging to the surfaces of foods or other materials can occur. In recent years, there has been renewed interest in understanding the potential 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. The concentrations that can be expected in most food products and non-plasticized food contact materials are several orders of magnitude lower than the concentrations that can be found in plasticized PVC. The significantly different concentrations require different methodology for their extraction and detection. Due to the widespread use of plasticized PVC in many commercial applications, background concentrations of phthalates are a concern when doing laboratory analyses. A liquid-liquid extraction with dichloromethane and hexane was used to extract phthalates from packaging. The extracts were then analyzed using a GC-MS/MS. Accuracy data showed spiked recoveries ranging from 71-124% in representative packaging. Phthalate concentrations in several different non-PVC printed and unprinted packaging will be presented. This data will help provide the Agency important information on potential phthalate exposure to consumers via food consumption.

P-14 Analytical method development and validation for the analysis of pesticides in flax using QuEChERS and lipid-removing dSPE

Jules C. Carlson, Daniel Bockru, and Sheryl A Tittlemier;

Canadian Grain Commission, 303 Main Street; Winnipeg, MB R3C3G8, Canada; jules.carlson@grainscanada.gc.ca;

Flax is an important crop with many uses for food or feed purposes, and provides many health benefits. QuEChERS techniques are commonly used for the analysis of pesticides in many agricultural products, but there are few methods for analysis in flax. Analysis of flax can be challenging due to the ~40 % lipid content, as lipids are non-volatile and can quickly dirty GC liners, GC/MS sources, and columns. A method was developed for the analysis of 15 pesticides in flax using QuEChERS with a lipid-removing SPE step with analysis by GC-MS. The efficacy of QuEChERS methods was assessed when using three different dispersive SPE (dSPE) cleanup steps: (1) a traditional Phenomenex dSPE with MgSO₄, PSA, and C18 step, (2) Agilent's Enhanced Matrix Removal - Lipid (EMR-Lipid) removal dSPE step, and (3) both dSPE steps from (1) and (2). Peak areas were measured for each pesticide after blank flax was fortified at 50 ug/kg of each pesticide and extracted. Area counts were largest and background interference was smallest when only the EMR-Lipid dSPE was used. Internal standard corrected mean recoveries for the final method ranged from 69 to 120 % with RSDs below 12 %. Limits of quantitation in flax were determined to be 10 to 50 µg/kg. The EMR-Lipid cartridge shows promise for cleanup of high lipid grains for pesticide analysis for GC/MS methods.

P-15 Multiclass Method for the Quantitation and Confirmation of over 120 Veterinary Drugs in Game Meat (Bison, Deer, Elk, and Rabbit) via Three Separate Methods using Rapid Polarity Switching Liquid Chromatography-Tandem Mass Spectrometry (LC-MS/MS)

Christine R. Casey¹, Wendy Andersen², Tara J. Nickel¹, Nicole T. Williams¹, and Patrick R. Ayres¹

¹Denver Laboratory, U.S. Food and Drug Administration, Denver, CO 80225

²Animal Drugs Research Center, Food and Drug Administration, Denver, CO 80225

The European Union (EU) conducted an audit in 2010 of the United States FDA programs designed to monitor pesticide residues, chemotherapeutic agents, industrial contaminants, and toxic elements in domestically produced animal derived foods. In order to address differences between the EU and FDA residue analysis programs for veterinary drug monitoring, the Denver Laboratory (DENL) developed a multi-class, multi-residue analysis program to qualitatively and quantitatively analyze domestic game meats (bison, deer, elk, rabbit) for over 125 compounds from veterinary drug classes including:

thyreostats, steroids, resorcylic acid lactones, beta-agonists, nitroimidazoles, amphenicols, tetracyclines, beta-lactams, fluoroquinolones, macrolides, sulfonamides, anthelmintics, sedatives, anti-inflammatory drugs, coccidiostats, and avermectins. The combined analytical program permits each game meat sample to be extracted and analyzed by the three independent methods for a combined analysis of 125 veterinary drug residues at regulatory required levels typically in the low part-per-billion range. The analytical program efficiently replaced ten separate analytical methods performed under contract by a third-party laboratory, and expanded the residue testing capability of the FDA DENL. To date, the DENL has analyzed over 200 regulatory game meat samples each for 125 veterinary residues. This poster will describe the development and validation for the qualitative and quantitative of all three methods.

P-16 LC-MS/MS Determination of Gyromitrin in Mushrooms as the Method to Identify False Morel Mushrooms

Narong Chamkasem

Southeast Food and Feed Laboratory (SFFL), **U.S. Food and Drug Administration**, 60 Eighth Street, N.E., Atlanta, GA 30309; USA, narong.chamkasem@fda.hhs.gov

False morel mushrooms have been responsible for severe intoxication, vomiting, diarrhea, jaundice, convulsions, coma and even death. Gyromitrin is the major toxin contained in these fresh mushrooms. The current method to determine gyromitrin in false morels is by physical examination. This method needs extensive technical training and there is no longer an expert available at FDA. FDA needs a more modern chemical testing method to accurately determine gyromitrin in the false morels to support the FDA regulatory program. Acetonitrile extraction and salting-out sample cleanup method was used to extract gyromitrin spiked into three different blank mushrooms. The sample extract was directly injected to an LC-MS/MS instrument to determine gyromitrin in the sample. Retention time against the authentic standard and at least two MRM transitions were monitored to achieve true positive identification of gyromitrin in the sample. The average recovery for gyromitrin at 0.4, 4, and 40 µg/g (n = 18) ranged from 81-106%, with a relative standard deviation of ≤ 8%. This method may be used to replace the physical examination technique to identify false morel mushrooms.

P-17 An Overview of the Canadian Food Inspection Agency's Chemical Residue and Contaminant Monitoring Programs

Faith Chou, John Moisey, Aaron Price, Leigh Miller, Beata Kolakowski

Canadian Food Inspection Agency, 1400 Merivale Road; Ottawa, ON K1A 0Y9, Canada; faith.chou@canada.ca

The Canadian Food Inspection Agency (CFIA) is responsible for monitoring chemical residues and contaminants in food to ensure that the food supply is safe and compliant with Canadian standards. The National Chemical Residue Monitoring Program (NCRMP), Food Safety Oversight (FSO) Program and Targeted Surveys are key monitoring programs developed and implemented by the CFIA. An overview of the program details, results and compliance rates will be presented. The NCRMP is used to demonstrate equivalence to international residue monitoring programs, to verify compliance with Canadian maximum residue limits and to identify trends and effectiveness of policies and programs. This program focuses on primary commodities such as meat, egg, dairy, fresh fruit and vegetables, processed products, honey and maple with samples collected from federally registered establishments and importers. The FSO Program was created to fill data gaps in non-meat commodity monitoring such as fresh fruit and vegetables, fish and seafood and manufactured food samples from retail stores. Targeted Surveys provide information about chemical hazards in manufactured and processed foods from retail stores that are not part of routine monitoring programs. These programs and surveys test more than 34000 samples and perform over 200000 tests annually for veterinary drug residues, pesticides, metals, contaminants, food colours and allergens. Positive samples are assessed against Health Canada's regulatory limits and non-compliant samples could result in follow-up actions including increased monitoring or food recalls.

P-18 Utilizing Innovative GC Liner Technology for Low-Level GC-MS/MS Pesticide Analysis

Angela Smith Henry¹, Amanda Kaspick¹, and Wei Song¹

¹**Agilent Technologies, Inc.**, 2850 Centerville Road, Wilmington, DE 19808, USA; angela.smith@agilent.com

Pesticides can be sensitive to active sites in gas chromatography-tandem mass spectrometry (GC-MS/MS) systems, which makes an inert flow path critical; however, some food matrices can foul the flow path. Therefore, inlet liners packed with glass wool are utilized to protect the GC-MS/MS system, even though glass wool can introduce active sites, since an irregular surface may not be fully deactivated or new active sites can open at breakage points of the glass wool. An alternative to glass wool liners, that still provides a barrier and volatilization site, has been developed and tested with low level pesticides in food matrices by GC-MS/MS. A set of approximately 20 pesticides was chosen to test with 7 matrices: strawberry, plum, onion, bell pepper, orange, avocado and spinach. Matrix matched calibration curves were generated with sandwich injections of matrix and pesticide standards at each concentration level. Sandwich injections were also employed to study signal degradation and liner consistency over repeated injections of the matrices, where 10 replicate injections of each matrix were completed in random order of matrix. Internal liner repeatability for each matrix was under 20% RSD for each of 7 liners. Liner reproducibility was evaluated using %RSD of response factors; the average across the compounds was under 20% for all matrices, except onion with a 22.2% RSD. The alternative liners are also compared to wool liners to determine efficacy and similarities or differences of liner performance.

P-19 Screen Unprepped Food Samples in 30-60 Seconds by GC/MS

Philip L. Wylie¹ and Melissa Churley²

¹Agilent Technologies, Wilmington DE

²Agilent Technologies, Santa Clara CA

Screening by GC/MS normally requires sample preparation such as QuEChERS or other liquid extraction methods. A simple and fast screening analysis that requires no sample preparation is demonstrated with the Quick Probe, a unique direct insertion GC/MS system. Compound identification of sample constituents is achieved through library searches when using a single quadrupole mass spectrometer in scan mode. A variety of food sample types have been studied including various oils, milk samples, spice mixes, beverages and flavorings. A 1.5 m high temperature capillary column was installed in a Quick Probe unit, which was mounted on a gas chromatograph. The column temperature was ramped quickly (maximum rate = 900 °C/min) leading to good chromatographic separation in less than 1 minute. Direct sampling was performed with solid glass probes held within a probe holder. The data were analyzed using MassHunter Unknowns Analysis software to perform library searches using the NIST library. Characteristic components within each matrix such as 5-hydroxymethylfurfural, vitamin E, thymol, piperanal, cis-vaccenic acid and 24-methylenecycloartenol (the latter two in olive oil) were determined with NIST match scores ranging from 76 to 99.5. The fast analysis of foods did not require sample preparation and allowed for a simple workflow to expedite screening. The technique has been successfully employed in the targeted analysis of drugs of abuse and is currently being evaluated in SIM mode for the targeted analysis of pesticides residues in foods.

P-20 Veterinary Drug Detection in Pork and Milk Using an Ultivo LC/TQ with ESI Ion Source

Theresa Sosienski and Karen Yannell

Agilent Technologies, Inc, 5301 Stevens Creek Blvd., Santa Clara, CA 95051, USA; theresa.sosienski@agilent.com;

Veterinary drugs aid in disease prevention and growth promotion for livestock operations. If improperly used, these drugs could accumulate in animal tissues and other animal-derived foods. In order to minimize this risk, global regulatory agencies set maximum allowable levels for many veterinary drugs in animal-derived products. Demonstrated here is the precise quantification of 12 regulated veterinary drug compounds in pork and milk. Samples of 2g of pork or milk were spiked with a mixture of veterinary drugs and prepared with a simple and quick extraction using acetonitrile, water, and Captiva EMR-Lipid SPE clean-up prior to analysis. Compounds of interest were separated in a 10-minute liquid chromatography method and analyzed using dynamic multiple reaction monitoring (dMRM) mode on the Ultivo LC/TQ equipped with an ESI source. All veterinary drugs could be accurately quantified at ½ the maximum residue limit (MRL), as defined by global veterinary drug regulations, while most could be quantified at 1/10 MRL, the lowest level tested in this study. All veterinary drugs showed excellent precision at the quantitation limit with RSD% below 14% for all compounds tested. Good linearity was also observed, with all calibration curves having R² values greater than 0.98. Recovery of the veterinary drugs ranged from 60-120% for most compounds, demonstrating the exceptional efficiency of the extraction method.

P-21 Analysis of Cannabinoids by Liquid Chromatography/Quadrupole-Time-of-Flight Mass Spectrometry (LC/QTOF-MS) with Accurate Mass

Imma Ferrer¹, E. Michael Thurman¹, Robert E. Sievers¹, and Jerry A. Zweigenbaum²

¹University of Colorado, Boulder, Colorado, and ²Agilent, 2850 Centerville Road, Wilmington, DE, 19808 USA j_zweigenbaum@agilent.com

We have analyzed 17 cannabinoids and 4 deuterated cannabinoid standards as part of a study on the detection and identification of these compounds in water, plants, and other products. Our first goal was to develop a method to detect and identify these compounds with liquid chromatography and accurate mass using time-of-flight analysis. This was accomplished with a slow gradient using acetonitrile and water with 0.1% formic acid and electrospray positive ionization. The second goal was the quantitation of cannabidiol (CBD) in various media (samples), which was carried out using deuterated standards and isotope dilution into water or plant extracts. Thirdly, the goal was to determine the accurate mass fragmentation patterns of these cannabinoid standards in order to better understand their chemistry and to have the ability to determine new cannabinoids, if they are detected in various plant samples. This was completed with MS-MS analysis using accurate mass. An example of bottled drinking water will be shown where we detected cannabidiol directly in the water sample without prior derivatization or treatment. Finally, the role of accurate mass will be discussed as an important tool in cannabis research.

P-22 Analysis of Terpenes and Cannabinoids by Gas Chromatography/Mass Spectrometry (GC-MS)

Imma Ferrer¹, E. Michael Thurman¹, Robert E. Sievers¹, and Jerry A. Zweigenbaum²

¹University of Colorado, Boulder, Colorado, and ²Agilent, 2850 Centerville Road, Wilmington, DE, 19808 USA j_zweigenbaum@agilent.com

We have analyzed 21 terpene standards and cannabidiol and cannabidiol acid as part of a study on the detection and identification of these compounds in hemp plants and other products as part of a research project on hemp and cannabidiol (CBD). Our first goal was to develop a method to detect and identify these terpene and cannabinoids with gas chromatography/mass spectrometry (GC/MS). We used a microfluidics GC/MS (named Intuvo) with a guard-chip design. The purpose of the guard chip is to protect the analytical column when plant extracts are analyzed directly, as will be shown in this poster. The second goal was to develop a simple extraction procedure for hemp plants using solvents compatible with GC/MS. This was accomplished with simple grinding and extraction with ethylacetate or methanol. Thirdly, the goal was to determine the fragmentation patterns of these terpene and cannabinoid standards in order to better understand their chemistry and to have the ability to determine new terpenes, if they are detected in various plant samples. An example of a hemp plant extract will be shown and the challenges of separating and identifying the mixture of isomers associated with the terpene family of compounds. Also data will be shown concerning the inject port degradation of cannabinoids. Finally, the role of GC/MS will be discussed as an important tool in cannabis research.

P-23 Quantification of Acrylamide in a Variety of Food Matrices by LC-MS/MS Triple-Quadrupole

Hui Zhao, Andre Santos, Jerry Zweigenbaum

Agilent, 2850 Centerville Road, Wilmington, DE 19808, USA; j_zweigenbaum@agilent.com

Acrylamide has been discovered to be present in cooked food, especially in heat-processed products such as potato chips and roasted and bakery products. Therefore, acrylamide content is commonly monitored in certain processed foods. In this study, a fast, high-throughput, and sensitive UHPLC-MS/MS method for the identification and quantification of acrylamide in a variety of food sample matrices was presented, using an Agilent 1290 Infinity II LC coupled to an Agilent 6470A triple quadrupole LC/MS system with Agilent MassHunter workstation software. The sample preparation consists of dispersing the sample in water followed by acetonitrile, partitioning the aqueous and organic layers by the addition of 4:1 (w:w) MgSO₄ : NaCl, diluting or drying down/reconstituting (according to the requirement of limit of quantification) a portion of the acetonitrile layer into water, filtering and then analyzing by LC/MS using positive electrospray ionization

mode. The isotopically labeled internal standard acrylamide-13C3 is added to compensate for matrix effects. The method was applied to raw potato, potato chips (three different brands), processed black olives, raw and roasted salted almonds, raw hazelnut, roasted salted pistachios and ready-to-drink coffee. The calibration range is from 0.1 to 200 ng/mL. The limits of quantification (meeting acceptable recovery, precision, and signal-to-noise (S/N) criteria) on matrices was achieved at 2.5 ng/g. Method criteria for data acceptance were established.

P-24 Simplified and Fast Analysis of Per- and Poly-fluoroalkyl Substances in Non-Potable Waters Using LC-MS/MS Triple-Quadrupole

Hui Zhao, [Jerry Zweigenbaum](#), Andre Santos

Agilent, 2850 Centerville Road, Wilmington, DE 19808, USA; andre_santos@agilent.com

There have been growing concerns about the use of per- and polyfluorinated alkyl substances (PFASs) due to their detection in all environmental media including air, water, and soil. Persistent chemicals, they have the potential to accumulate in the environment and impact the food chain, affecting fish, birds, livestock, and humans. Detection of PFASs at parts per trillion (ppt) levels is often required. This study describes a method for the separation and detection of 28 per- and polyfluorinated alkyl substances (PFASs) in water samples. The method uses an Agilent 1290 Infinity II LC coupled to an Agilent 6470A triple quadrupole LC/MS system with Agilent MassHunter workstation software. All the PFASs included in the ASTM 7979 method are analyzed using the same sample preparation protocol described. Water samples of 5 mL are diluted with an equal volume of methanol and injected directly for a reporting limit of 10 parts per trillion (ppt, ng/L) or lower for most of the compounds. The method was evaluated in water and quantified with external standards, showing satisfactory results, including identification, selectivity, linearity, reporting limits, accuracy and precision. In addition, this method can be used for the simultaneous detection and quantification of PFAS residues in reagent, tap, surface, ground and wastewater matrices.

P-25 SPME Arrow Sampling of Terpenes in Cannabis Plant Material

Jessica Westland, **Agilent**, 2850 Centerville Rd. Wilmington DE 19808, USA

With the growing legalization of medicinal cannabis worldwide, methods for qualifying and quantifying terpene concentrations has risen to the forefront in the analytical industry. The development of faster and more efficient method that will produce rapid and accurate results at a low cost is highly desirable. Since terpenes have high vapor pressures, and are extremely volatile, they are excellent candidates for static headspace gas chromatography (GC) analysis. PAL SPME Arrows can be used for both qualitative and quantitative determination of terpenes in plant material by headspace (HS) sampling combined with GC/MS. This approach offers several advantages compared to solvent extraction and GC-FID. It does not require the use of organic solvents, does not coextract matrix (which could potentially interfere with the chromatographic analysis or contaminate the GC system), and provides additional means of peak identification and purity using spectral data. PAL SPME Arrows provided the sensitivity and robustness needed to profile the predominant terpenes in an unknown variety of cannabis plant samples.

P-26 Optimizing Sample Preparation in Pesticides Analysis for Cannabis

[Jessica Westland](#)¹, [Anastasia Andrianova](#)¹; [Pete Stone](#)²

¹**Agilent**, 2850 Centerville Rd. Wilmington DE 19808 (USA);

²**Agilent**, 5301 Stevens Creek Blvd, Santa Clara, CA 95051 (USA)

At the present time, many U.S. state-regulated pesticides list for cannabis can be analyzed exclusively by LC/MS/MS. Notable exceptions include California, Florida, and Nevada, where GC/MS/MS is also required. The states requiring GC/MS are expected to grow as more compounds and lower detection limits are required. In this work, the detection and quantitation of all LC amenable pesticides and mycotoxins were reliably met by at least 50% of the current California legislative safety action limits in cannabis dried flower samples (LODs range between 0.5 – 50 ppb; Malathion's LOD = 100 ppb). Forty-three GC-amenable pesticides regulated by the Bureau of Cannabis Control in California met the

established limits of quantitation (LOQs) with the Agilent 8890 GC combined with an Agilent 7010B triple quadrupole GC/MS system.

The Agilent standardized sample preparation procedure aligned with Agilent's multiplatform approach provides a rapid return on investment (ROI) and a stable foundation to meet current and future testing requirements.

P-27 Screening of pesticide residues in food by using high-throughput GC-MS/MS with fast GC conditions

John Dane,¹ Junkei Ko,² and Kiyotaka Konuma ²

¹JEOL USA, Inc., 11 Dearborn Rd.; Peabody, MA 01960, USA; jdane@jeol.com;

²JEOL LTD; Akashima, Japan

With a growing concern about food safety, high throughput screening of pesticide residues is more important than ever. Speeding up the analysis using Fast GC methods is one option that can help with this. In this work, we narrowed the conventional GC method of 40minutes to measure all 266pesticides to a Fast GC method that required only 15minutes to complete. A 10ppb sample mixture was used to check the reproducibility of each pesticide (n=8). Although several peaks overlapped in the TICC when using the fast GC conditions, good reproducibility was observed in the SRM chromatograms. This indicates that the speed for switching SRM channels is sufficient for the fast GC conditions. The duration for each SRM channel was limited to only 2ms in the busiest time segment. However, the SRM capability of 1000transitions/second made it possible to change SRM channels to less than 2ms. The CV was evaluated for all 266pesticides in the 10ppb mixture. We found that 90% of the pesticides had a CV with less than 20%, and 59% of the pesticides had a CV with less than 10%. These results were due to the high sensitivity realized by ion accumulation in the collision cell and then their ejection as a pulse to the detector. The ion current is only measured during the pulse, thus eliminating the noise from the intervals between the pulses. The results presented will show that the JMS-TQ4000GC with fast GC conditions can be used for screening pesticides on food at the 10ppb MRL.

P-28 Vacuum Assisted Sorbent Extraction (VASE) Thermal Desorption-GC-MS: A Robust, Solvent-Free Technique for Chemical Residue Analysis

Sage J. B. Dunham, Victoria L. Noad, Bailey S. Arakelian, Daniel B. Cardin

Entech Instruments, Inc, 2207 Agate Court, Simi Valley, CA 93065, USA; sagedunham@entechinst.com;

Static headspace extraction techniques such as solid phase microextraction (SPME) harbor considerable promise because of their capacity for capturing volatile and semi volatile organic compounds (VOCs & SVOCs) from diverse sample compositions (i.e., solids, liquids, & gases), the simple sample preparation requirements, and the ease with which the techniques can be implemented. Despite this promise, the utility of headspace extraction techniques is hampered by a limited chemical coverage (lower volatility SVOCs are difficult to detect), a poor dynamic range for multi-analyte quantitation (limited loading capacities dictate that abundant analytes must compete for active sites in the sorbent bed), and, in some instances, a general dearth of analytical rigor. In this work we describe a new technique for static headspace extraction – vacuum assisted sorbent extraction (VASE) – and the application of this technique to examine the chemical profile of a wide assortment of liquid and solid samples. In contrast to existing static headspace extraction techniques – which rely on heat, agitation, and solution chemistry to promote analytes into the headspace – VASE also utilizes the combination of a large sorbent phase and a reduced pressure extraction environment to expand chemical coverage and improve sensitivity. Example applications are shown in the areas of foods and beverages (e.g., milk, cheese, meat, alcoholic spirits, coffee, produce), lab consumables (e.g., nitrile gloves, O-rings, pump oil, vacuum grease), environmental samples (e.g., ground water, soil), cannabis consumer products (e.g., edibles, flower), and forensics (e.g., drug and accelerant residue, fingerprints). When combined with thermal desorption GC-MS, VASE is shown to be an exceptional tool for quantitative and qualitative analysis of both native compounds and residual adulterants.

P-29 Trace-Level Quantification of SVOCs in Water via Vacuum Assisted Sorbent Extraction (VASE) Thermal Desorption-GC-MS

Sage J. B. Dunham, Victoria L. Noad, Daniel B. Cardin

Entech Instruments, Inc, 2207 Agate Court, Simi Valley, CA 93065, USA; sagedunham@entechinst.com;

Current analytical methods for evaluating volatile and semi volatile organic compounds (VOCs & SVOCs) in water often necessitate large sample volumes (1 L or more), are labor intensive, and require liquid-liquid extraction into organic solvents. Here we present a sensitive, solvent-free method – termed vacuum assisted sorbent extraction (VASE) – for extraction and pre-concentration of VOCs and SVOCs in preparation for thermal desorption-GC-MS. In VASE, the sample (often less than 1 mL for water and 1 g for solids such as soil) is evacuated in the presence of a headspace sorbent pen (HSP), which is a vacuum-tight cartridge containing sorbent. In combination with optional heat and agitation, reduced pressure helps promote compounds into the headspace where they adhere to the sorbent bed. Following VASE, the HSP is thermally desorbed directly onto a GC-column for analysis. VASE and the sorbent pen combine the large sorbent capacity and durability of thermal desorption tubes with the ease of use and in-vial extraction capabilities of solid phase microextraction (SPME) to create a robust and highly sensitive headspace extraction approach. The application of VASE for trace-level quantitation of contaminants in water is presented. Specifically, we demonstrate the extraction and quantification of compounds with boiling points ranging from 80°C to over 550°C, including 2-6-ring polyaromatic hydrocarbons, phenols, pesticides, chlorinated hydrocarbons, and disinfection by-products from water. Limits of quantification as low as 1 ng/L are demonstrated for 50 mL of sample and 500 ng/L for 1 mL of sample. These results show that, when combined with TD-GC-MS, VASE is a highly sensitive and robust technique for TRACE-level analysis of contaminants in water.

P-30 Method for Simultaneous Determination of 57 Pesticide Residues in Barley, Malted Barley and Sweet Wort Using the LC-HR-MS/MS Technique and Its Application for the Study of Fade of Pesticide during Malting and Brewing Processes

Martin Dušek,¹ Sylvie Běláková,² Karim Cristina Piacentini,³ Alexandr Mikyška,¹ and Jana Olšovská;¹

¹Research Institute of Brewing and Malting, Lípová 15; Prague, CZ-120 44, Czech Republic; dusek@beerresearch.cz;

²Research Institute of Brewing and Malting, Mostecká 971/7; Brno, CZ-614 00, Czech Republic;

³University of Sao Paulo, Biotechnology Department, Av. Professor Lineu Prestes, 2415, Sao Paulo, Brazil

Malt is the main raw material for beer brewing. The composition and quality of malt directly affect the flavor and quality of beer. During the growing season, various agrochemical sprays may be used to ensure high quality and food safe crop. The pesticides remaining on plant represent a potential source of unwanted contamination during brewing beer. Twenty four samples of malted barley originating from Brazil harvested from three locations: Taquarivaí (São Paulo), Passo Fundo and Vitor Graeff (Rio Grande do Sul), were used for this study. Fates of organophosphorus insecticides (chlorpyrifos and pirimiphos-methyl), morpholine fungicide (fenpropimorph), strobilurin fungicides (pyraclostrobin and trifloxystrobin) and triazole (tebuconazole) fungicides from barley to malt and from malt to sweet wort were determined. Pesticide residue analysis was carried out by LC/HR-MS/MS. The citrate buffered QuEChERS (EN 15662) sample preparation method was used for extraction of pesticide residues from grain matrices and provided good analytical results for target pesticides. An additional clean-up on PSA (Primary-Secondary Amine) SPE columns in combination with solvent exchange was necessary prior to trace analysis of target pesticide residues in the samples of sweet wort. Pesticide residues in malt were significantly lower than in barley. The amount of remaining pesticides after malting ranged from 10% to 80% for pirimiphos-methyl and fenpropimorph, respectively. All studied pesticides have high log P values (>3) and so tended to remain mainly in spent malt after mashing. The carryover of pesticide residues from malt to sweet wort after mashing therefore ranged from 5% to 15%.

P-31 A comprehensive evaluation of different approaches for the determination of highly polar pesticides in food matrices

Sonia Herrera López, Barbara Kiedrowska, Jos Scholten, André de Kok

WFSR – Wageningen Food Safety Research, National Reference Laboratory (NRL) for Pesticide Residues in Food and Feed, Wageningen, The Netherlands; sonia.herreralopez@wur.nl; andre.dekok@wur.nl

Non-selective highly polar herbicides are well known, especially glyphosate, because of their high effectiveness and low costs. Glyphosate, together with many of the target polar pesticides and plant growth regulators included in this evaluation are some of the most frequently used pesticides worldwide.

Their physical-chemical properties make their analytical determination a major challenge for the analytical chemists. Although no adverse effects have been observed on humans, these compounds and their metabolites, which have sometimes similar toxicological characteristics, have been detected in water, soils and crops. However, due to the difficulty of the analysis of high polar compounds with old procedures, there is not much information available about the occurrence of these compounds. Maximum residue levels (MRLs) have been established for fruits, vegetables and cereals by the European Union and other countries. In some cases, the lack of information about the metabolites and/or degradation products have caused residue definitions to be incomplete.

Recently, a wide range of methodologies have been proposed trying to make the analysis of polar compounds successful. Unfortunately, retention time shifts, high matrix effects and rapid degradation of the used columns, limit their robustness, utility and their cost-effectiveness.

The aim of this study is the evaluation of a methanol-based extraction method with a clean up step¹. For the clean up step, several sorbents from different brands (EMRd, CAPTIVA, Oasis HLB, Oasis WCX and MCX), with diverse action mechanism, have been tested. In addition, three different UHPLC columns have been assessed during this evaluation, in order to determine which one provides the best results in terms of chromatographic separation, peak shape and sensitivity. Besides these tests, also different dilution factors for the final extract were applied in order to check the impact on the matrix effect.

The UHPLC-MS/MS experiments were performed with a UHPLC (Nexera LC) coupled to a hybrid quadrupole/linear ion trap mass spectrometer system (Sciex 6500+ QTRAP[®]). Isotopically labeled internal standards for all compounds (except for N-acetyl-AMPA) were used for recovery and matrix effect correction, although real (no corrected) recoveries were also calculated to test the effective extraction yields.

The results of the evaluation study and validation data of the optimal method for 15 target compounds will be presented.

P-32 Irvine Rapid Analytical Method (IRAM): A Sensitive Method for the Analysis of Pesticide Residues in High Fat Food Products

Madison Hanson, Eugene Chang, Janet Blount, Irene Cassias, Darin Files, Shannon Lane, Andrew Limson, Sarah Wilson, Jacqueline Sram, Olusegun Ajayi, and Kim Thomas-Cruse

U.S. Food and Drug Administration, Pacific Southwest Food and Feed Lab, Irvine, CA 92612

Pesticide analysis requires a reliable and dependable multiresidue method with the ability to cover various matrices. This has been problematic for high fat commodities because of the difficulty to distinguish the fat from pesticides on the sorbent. The Irvine Rapid Analytical Method (IRAM) is a simple extraction process that combines the ease of dispersive solid phase extraction (d-SPE) and sensitivity of previously established methods. The authors conducted a method validation to demonstrate that the IRAM could successfully analyze high fat matrices on both LC-MS/MS and GC-MS/MS platforms. This study would enable pesticide scientists to utilize one extraction method to analyze approximately 300 pesticides on two platforms; significantly reducing the resources required for the detection of pesticides in a high fat matrix. The method validation included a nut, oil, and fish fortified at three different levels in replicate. Although some outliers were observed contingent upon the matrix, validation data was within acceptable range set forth by the FDA Pesticide Analysis Program guideline. Additionally, an international proficiency test was performed to demonstrate method validity and accuracy. Results of this study confirm that the IRAM can successfully be utilized on both LC and GC platforms.

P-33 Study for Residue Analysis of Pinoxaden and its metabolites in Food Matrices

Ji Young Kim,¹ Ji Young song,¹ A Reum Han,¹ Nuri Seong,¹ Jong Soo Kim,¹ Sang Soon Yoon,² Yong Hyun Jung,² Jae Ho Oh² and Hyochin Kim¹

¹ **Ministry of Food and Drug Safety-Seoul Region, Division of Import Food Analysis**, Seoul 07978, Korea, jykim98@korea.kr

² Pesticide and Veterinary Drug Residues Division, National Institute of Food and Drug Safety Evaluation, Ministry of Food and Drug Safety, Cheongju 28159, Korea;

Pinoxaden is the phenylpyrazoline herbicide developed by Syngenta Crop Protection, Inc and marketed on 2006. Although it is not registered in Korea, maximum residue level for wheat and barley were set by import tolerance. Thus, MFDS official analytical method determining Pinoxaden residue was necessary in various food matrixes. Satisfaction of international guideline of Codex (Codex Alimentarius Commission CAC/GL 40). In this study, Liquid Chromatography-Tandem Mass Spectrometry (LC-MS/MS) method was investigated to analyze residue of Pinoxaden and its metabolites (M2, M4 and M6) in foods. Pinoxaden was extracted with acetonitrile (w/ 1% formic acid), partitioned and concentrated with anhydrous magnesium sulphate and sodium chloride. To remove the interferences, anhydrous magnesium sulphate and PSA was performed before LC-MS/MS analysis. Five agricultural commodities (mandarin, potato, soybean, hulled rice, and chilli pepper) were used as group representative to verify the method. The liner matrix-matched calibration curves were confirmed with coefficient of determination (r^2) > 0.99 at calibration range 0.0025-0.25 mg/kg. The limits of detection and quantification were 0.01 mg/kg, which is the threshold of Positive List System (PLS). Mean average accuracies of Pinoxaden, M2, M4 and M6 were shown 89.9-113.1, 82.1-111.8, 84.2, 116.8 and 73.6-108.7 %. The precision of Pinoxaden and its metabolites was also shown less than 8.3% for all five samples. The method investigated in this study is suitable to Codex and MFDS guideline for residue analysis. Thus, this method can be useful for determining the residue in various food matrixes in routine analysis.

P-34 Development of a simultaneous multi-residue analysis for screening and confirmation of 6 veterinary drugs in chicken muscle by liquid chromatography tandem mass spectrometry

MinKyoung Kim¹, JunYoung Choi¹, NamSun Kim¹, HeeSun Kim¹, JongWon Kim¹, HuiSeung Kang², ByeongHoon Cho² and JaeHo Oh²

¹Hazardous Substances Analysis Division, Seoul Regional Office of Food and Drug Safety, Seoul 07978, Republic of Korea

²Pesticide and Veterinary Drug Residues Division, National Institute of Food and Drug Safety Evaluation, Ministry of Food and Drug Safety, Cheongju 28159, Republic of Korea; mg6153542@korea.kr

A simple and fast analytical method based on liquid chromatography tandem mass spectrometry was developed for detection of the veterinary drugs acetanilide, anthranilic acid, antipyrine, diphenhydramine, DL-methylephedrine and phenacetin in chicken muscle. The target analytes were extracted from sample using acetonitrile followed by clean-up with C18 and liquid-liquid purification with saturated n-hexane. A reverse-phase analytical column was employed with a mobile phase comprising (A) 0.1 formic acid in distilled water and (B) 0.1% formic acid in Acetonitrile to achieve the best chromatographic separation. Matrix-matched calibration curves ($r^2 \geq 0.9995$) were constructed using six concentration of 1, 2, 5, 10, 20 and 40 $\mu\text{g}/\text{kg}$ in chicken muscle matrix. Recoveries at three spiking levels (5, 10 and 20 $\mu\text{g}/\text{kg}$) ranged 84.1 - 103.3% with intra-day and inter-day relative standard deviation (RSD) of $\leq 10.4\%$. The calculated limits of quantification (LOQ) were 1.1 - 6.0 $\mu\text{g}/\text{kg}$.

P-35 Doing More with Less

Louis C. Mayer, Syngenta Crop Protection, LLC, 410 Swing Rd.; Greensboro, NC 27409, USA; louis.mayer@syngenta.com

Current strategy in industry, academia and government agencies is to provide analytical methods to increase throughput by decrease of sample size, extraction time and sample analysis time for single and multi-compound requirements. The capability to increase sample throughput provides faster data generation. Having access to data faster translates into better efficiency for critical decisions on study design, changes in workflow and project management. An overall increase in sample throughput benefits the CRO and sponsor by allowing more studies to be completed within an established timeframe allowing more control of time management for planning other studies and/or submissions. Identifying bottlenecks, wastes, areas that can increase efficiency and application is crucial for migration from conventional methodology to HT methodology.

P-36 Optimization of Instrumental Conditions for Fast and Sensitive Analysis of Multiple Pesticide Residues with Low-Pressure Gas Chromatography

Sergio H. Monteiro,^{1,2} Steven J. Lehotay,² and Yelena Sapozhnikova²

¹Biological Institute, Environmental Protection Research Center; Sao Paulo; Brazil; monteiro@biologico.sp.gov.br;

²USDA Agricultural Research Service, 600 East Mermaid Lane; Wyndmoor, PA 19038, USA;

Nowadays the laboratories are increasingly looking for ways to speed up analysis time, combining as many analytes as possible into single multi-residue methods to increase sample throughput and reduce analysis costs without compromising results. Low-pressure gas chromatography (LPGC) using a 5 m, 0.18 mm i.d. guard column and 15 m, 0.53 mm i.d. analytical column provides a fast separation with high signal/noise ratio in MS(/MS) detection. In this study using an Agilent 7010 MS/MS, we re-optimized injection parameters and column flow in LPGC-MS/MS to increase its sensitivity compared with previous instrumental conditions. We compared helium carrier gas flow rates (0.25 – 2.5 mL/min), injection modes (split vs. splitless), and three types of injection liners. Use of switched septum purge mode was also found to make a significant improvement in injection efficiency over the default setting of constant 3 mL/min. Unlike our previous LPGC-MS/MS optimizations using other instruments, we found that 1 mL/min gave substantially higher response than 2 mL/min. Ultimately, 1.5 μ L splitless injection into a low-pressure drop liner with glass wool of final QuEChERS extracts in acetonitrile containing analyte protectants gave about double the responses in relation to previous conditions using split mode of an equivalent amount of the same samples. The optimized conditions were applied in multiresidue analysis for >150 analytes with 3 ion transitions each in <10 min, which afforded use of 5-fold higher solvent/sample ratio during extraction to increase recoveries of many analytes and equal or improve upon previous LOQs.

P-37 LC-MS/MS Multi-Residue Method for Determination of Tetracycline, Sulphonamides and Amphenicols Residues in Bovine Milk

Sergio H. Monteiro, Biological Institute, Environmental Protection Research Center; Sao Paulo; Brazil; monteiro@biologico.sp.gov.br

Milk has a prominent place in human nutrition, providing essential vitamins and minerals for people of all ages, especially for children. Antimicrobial residues are considered the main chemical contaminants in milk and may pose a potential risk to the consumer health, as well as possibility of interfering in technological processes of dairy products. The present study describes a simple method that involves a deproteinization of milk samples using acetonitrile followed by ultrafiltration using the Captiva ND Lipids cartridges, for simultaneous determination of eight antibiotic residues in bovine milk. Validation procedure was based on criteria of the European Commission Decision 2002/657/EC considering the following parameters: selectivity, limits of detection (LOD) and quantification (LOQ), precision, accuracy, and recovery. Since the present study was designed to develop a simple and fast method for screening and quantifying antimicrobials in milk at the lowest possible levels, decision limits ($CC\alpha$) and the detection capacity ($CC\beta$) were not considered. The developed method was applied to 20 Ultra High-Temperature Pasteurization (UHT) milk samples collected from Sao Paulo local market in Brazil. The recoveries for the studied antibiotics ranged from 88–108% with relative standard deviations between 2 and 10%. The limits of quantification were below the maximum residue limits (MRLs), including for chloramphenicol with MRL = 0.3 mg/kg, which is banned in both countries the USA and Brazil. No residue was found in the analyzed samples. The developed method is fast, sensitive and suitable for analysis of the selected residues in milk.

P-38 Comprehensive Untargeted Screening and Quantitation of Pesticides in Cannabis Using GC \times GC and High Performance Time of Flight Mass Spectrometry

Todd S. Richards, David E. Alonso, Lorne M. Fell, Joseph E. Binkley

LECO Corporation, 3000 Lakeview Ave, Saint Joseph, MI, USA; Todd_Richards@leco.com

As states continue to institute laws for medical and recreational use of cannabis, ensuring available products are safe for public consumption has become a primary focus of various governmental agencies. While potency testing garners

the most public attention, pesticide quantitation remains the most difficult hurdle for laboratories to overcome. The complexity of varietal and product matrices are the greatest obstacle to efficient, accurate detection and quantitation of pesticides in cannabis. Untargeted screening approaches could be used to expand current pesticide lists in a meaningful, data directed way, or in the case of a health issue, to identify possible contaminants.

GC×GC/MS analysis of matrix matched standards was performed using a LECO Pegasus BT 4D high performance time of flight mass spectrometer (ToFMS). Data were acquired from m/z 45-600 at 200 spectra/second. Quantitation curves for each of the pesticides were created and used to quantify incurred pesticides in unspiked extracts. Additionally, non-target data processing was used to search for contaminants that are not included in the typical testing workflow. The improved chromatographic resolution afforded by GC×GC paired with high performance TOFMS significantly decreased matrix interferences with target compounds leading to improved quantitation accuracy and limits of detection. This improved chromatographic separation also positively impacted the detection and identification capability for non-target compounds.

P-39 What is Identification? Comprehensive Characterization of Exposome Samples via GC×GC-High Resolution TOFMS

Todd S. Richards, David E. Alonso, Lorne M. Fell, Joseph E. Binkley

LECO Corporation, 3000 Lakeview Ave, Saint Joseph, MI, USA; Todd_Richards@leco.com

Historically targeted analysis has been the only practical route to evaluate complex environmental samples. Focused testing, while effective may miss emerging or untargeted compounds. Recent improvements in detection and data processing capabilities allow scientists to more fully evaluate these same samples using non-targeted techniques. Often these evaluations are driven by a lab's available equipment and are heavily influenced by current workflows rather than an evaluation of multiple hardware & software platforms in the hands of expert users. The EPA is conducting a multiple lab, multiple platform evaluation for non-targeted analyte detection and identification. The goal of ENTACT (EPA's Non-Targeted Analysis Collaborative Trial) is to evaluate several platforms and strategies for analysis and reporting to facilitate future improvements to non-targeted analysis. Our previous work in this project was on the first ten blinded standard mixtures. This follow-up presentation will include results for spiked reference materials (household dust, serum and silicone wristband) and preliminary results from individual compound standards, being used to create a high resolution, accurate mass library. The increased chromatographic peak resolution achieved with GC×GC directly leads to increases in the number of unknowns found as well as increased match confidence. Deconvolved spectra were matched to existing commercial GCMS libraries and screened using a novel match ranking scoring system based on retention index value, molecular ion mass accuracy and fragment ion formula correlation. For compounds without NIST spectra, presumptive matches may be made based on molecular ion confirmation, predicted fragment mass accuracy and fragment isotopic ratios.

P-40 Novel UHPLC-MS-MS Method for Analysis & Quantitation of California Regulated Pesticides in Cannabis Oil Using Dual ESI and APCI Source

Jacob Jalali¹, Avinash Dalmia¹, Erasmus Cudjoe¹, Feng Qin¹, Ben Armstrong², Luke Ward²

¹**PerkinElmer**: 75 Nicholson Ln, San Jose, CA 95134

²Juniper Analytics LLC: 1334 NE 2nd St Bend, OR 97701

Cannabis oil is a concentrated extract from cannabis sativa plant. Pesticide analysis in cannabis oil, has been a challenge in past few months due to high percentage of cannabinoids in matrices and heavy ion suppression. Recent changes in regulatory guidelines for the quality control of medicinal cannabis and cannabis for recreational use has highlighted some interesting analytical challenges in the analysis of pesticides in cannabis oil. This study will detail an analytical UHPLC-MS-MS method for low ppb level quantitation of CA regulated pesticides in cannabis oil. It is anticipated that this study will help support method development efforts towards improved robustness and quantitative accuracy in this new and emerging industry. In this research, cannabis oil with spiked deuterated internal standards, is extracted with acetonitrile and after filtration, supernatant is transferred to HPLC vial for LC-MS-MS analysis. A mix of 20 deuterated internal standards were used across the run for correction of any matrix effect and achieving a good recovery for pesticides. PerkinElmer Quasar SPP Pesticides HPLC column (100x4.6mm, 2.7um) is used for separation of pesticides. Cannabis oil samples were prepared and analyzed at Juniper Analytics (Bend, OR). The UHPLC for this investigation was

a PerkinElmer LX-50. The MS/MS instrument was a QSight 220. Investigating mobile phase solvents, flow rates, injection volume, peak shapes, injection modes, ionization source parameters and cannabis strain-base product ion scans, were part of this research. Good accuracy, recovery and long-term stability were found on spiked oil samples, by using this analytical method.

P-41 Overcoming Challenges Associated with analysis of all of pesticide residues in cannabis, hemp and other derived matrices using LC-MS/MS

Avinash Dalmia¹, Erasmus Cudjoe², Jacob Jalali², Jeff Wu², Feng Qin²

¹ PerkinElmer, 710 Bridgeport Avenue; Shelton, CT 06484, USA; Avinash.dalmia@perkinelmer.com;

² PerkinElmer, Woodbridge, ON, CA.

Pesticide analysis in cannabis and hemp is very challenging since it is a dirty matrix due to its complex composition. Moreover, you need to measure pesticides in variety of cannabis derived matrices such as fresh plant, dried cannabis flower, hemp, oil, distillate, edibles and others. Different sample matrices lead to variable signal ion suppression and matrix interference. Also, pesticide analysis in cannabis and other food matrices is done by both GCMS and LCMS since some nonpolar and chlorinated pesticides such as pentachloronitrobenzene, chlordane, endosulfan and others are difficult to ionize with electrospray source used in LCMS systems. We would demonstrate how we were able to measure these compounds at low levels in cannabis using LCMSMS with APCI source. The ionization mechanism of chlorinated pesticides such as pentachloronitrobenzene, chlordane, endosulfan, etridiazole, chlorfenapyr and others in cannabis using LCMSMS system with APCI source would also be presented. Two different LCMSMS methods with electrospray and APCI source were used for low level analysis of 96 pesticides (including hydrophobic and chlorinated pesticides analyzed by GCMS) in cannabis, hemp and other cannabis derived matrices such as distillate, edibles and others. In this work, we used a generic extraction method with dilution, utilized 20 deuterated internal standards, selected the best MRM transitions and optimized the gradient to demonstrate low level analysis of pesticides with good recovery by compensation for ion suppression and less matrix interference in a complex cannabis matrix and other derived cannabis products.

P-42 Per and Polyfluorinated Alkylsubstances (PFAS) Analysis in Drinking Water, Sediments, and Food Samples by QuEChERS, SPE, and LC-MS/MS

Syljohn Estil,¹ Agustin Pierri,² Ramkumar Dhandapani,³ and [Scott Krepich](#);³

¹Sanitation Districts of Los Angeles County-San Jose Creek Water Quality Laboratory, Whittier, CA;

²Weck Laboratories, City of Industry, CA 91745;

³**Phenomenex, Inc.**, 411 Madrid Avenue; Torrance, CA 90501; scottk@phenomenex.com

Per and Polyfluorinated Alkylsubstances (PFAS) are a class of highly stable synthetic organic compounds used in a wide variety of industrial and commercial applications including surface treatment for textiles, packaging materials, non-stick cookware, and fire-fighting foams. PFAS are characterized by a hydrophobic fluorinated alkyl chain and a hydrophilic functional group. They are persistent in the environment due to the exceptional stability of the C-F bond. These have been detected throughout the global environment, food products, even human plasma. PFAS are associated with various adverse health effects, they are bioaccumulative, ubiquitous, and their analysis level requirements are very low to account for an expected lifetime of exposure. There are several methods available for the extraction and analysis of PFAS in aqueous samples. However, very few procedures are available for extracting these compounds in solid matrices such as sediments and food samples. Presented are three methods making use of various sample preparation techniques for the analysis of PFAS. The methods include, direct inject technique for drinking water, QuEChERS for sediment samples, and QuEChERS followed by SPE for food samples (milk, eggs, and fish tissue). All are validated LC-MS/MS procedures.

P-43 Single Column GC Solution for the Determination of Residual Solvents and Terpenes in Cannabis Matrices

Ramkumar Dhandapani, [Scott Krepich](#), Yuri Belov, and Rola Elabaji

Phenomenex, Inc., 411 Madrid Avenue; Torrance, CA 90501; scottk@phenomenex.com

While legalization of medical and adult-use marijuana continues to proliferate throughout North America, the use of cannabis remains illegal on a federal level in the United States. As such, the range of volatile contaminants, such as residual solvents, have diverse ranges of regulatory guidance on a state by state basis. In addition, terpenes account for the flavor and aroma of cannabis, profiling via GC-FID is a very important tool in identifying and quantifying terpenes in cannabis products for both quality and branding purposes. 624-type phases are common for pharmaceutical residual solvents due to their excellent selectivity for USP General Chapter <467> residual solvents, which are generally consistent with typical residual solvents to examine in cannabis and cannabis products. Provided is a method for both residual solvents and terpenes in one column. The sample has both light and heavy boiling analytes. Here we utilized the versatile 624 selectivity and high temperature limit of the Zebron ZB-624PLUS™ to provide a one column solution to cannabis residual solvent and terpenes testing via GC-FID.

P-44 Analysis of Mycotoxins in Cannabis Plant and Cannabis-containing Products

Sareeta Nerkar and Maria Ofitserova

Pickering Laboratories, Inc, USA; sareetan@pickeringlabs.com; 1280 Space Park Way, Mountain View, CA 94043

As medical and recreational Cannabis use gains broader acceptance, regulations are being put in place to mandate the testing of consumer products containing Cannabis. Legally available Cannabis plant and cannabis-containing edible products are tested for the presence of pesticides, heavy metals, residual solvents, and other harmful substances. State regulations have established maximum allowed levels for total Aflatoxins and Ochratoxin A in cannabis products, and laboratories are looking for methods to analyze these compounds.

We present an easy and sensitive method to analyze Aflatoxins B1, B2, G1, G2 and Ochratoxin A in cannabis plant and edible products. Mycotoxins are isolated using immunoaffinity clean-up columns and analyzed with fluorescence detection. To increase the sensitivity of Aflatoxins B1 and G1, an in-line photochemical reactor is installed before the detector. This method utilizes standard HPLC equipment and allows laboratories to easily determine Mycotoxins at levels below the limits established by state regulations.

P-45 Optimizing GC-MS and GC-MS/MS analysis of 3-MCPD and glycidyl esters

Jana Rousova, Joe Konschnik, Hansjoerg Majer, Alexandria Pavkovich Bush and Chris English

Restek Corporation, 110 Benner Circle, Bellefonte, PA, USA 16823, USA

3-MCPD and glycidyl esters in edible oils are contaminants that are formed through refining processes and several of these substances have been classified as possible human carcinogens. Methods, which are similar to one another, have been developed by ISO, AOCS, and DGF for analyzing these contaminants. While the methods cover extraction and derivatization techniques in detail, very little attention is paid to the GC-MS methods. With emerging automated systems, it is important to simplify and speed up the method by optimizing the parameters, to include switching to split injection.

The initial optimization of the temperature program led to an 8-minute decrease in the analysis time and additional time can be saved by utilizing free method development software. The employment of split injection resulted in better peak shape and achieved limits of detection that were comparable to splitless injection. Further evaluation of split injection revealed that the same performance is achieved regardless of inlet temperature resulting in greater flexibility for different inlet configurations.

P-46 SPE Disks for the analysis of perfluorinated compounds in large water volumes by LC-MS/MS

Jim Fenster¹, Kaynoush Naraghi², Corentin Germain², Michel Arotçarena², Raphaël Durand², and Sami Bayouhd²

¹AFFINISEP USA LLC, PO Box 620828, Middleton, WI 53562; jim.fenster@affinisep.com

²AFFINISEP SAS - Bd Sonopa – Pôle d'innovation des couronnes, 76650 Petit Couronne, France – contact@affinisep.com

Perfluorinated compounds (PFCs) are a family of molecules made of varying lengths of fluorocarbons chains and a functional group like carboxylic or sulfonic acids. PFCs have received worldwide attention in recent years due to potential human health effects. Perfluorinated compounds in water are usually analyzed using solid phase extraction (SPE) as described in EPA 537.1.

In order to improve the sensitivity and to accelerate the SPE process, we have developed SPE disks for the analysis of larger volume of water in a shorter time than classical cartridges SPE. SPE disks are thin, dense, soft, and uniform SPE membranes that allow the best interactions with analytes, without any channeling even with high flow rates.

In this poster, 5 perfluorinated compounds, with different lengths and chemistries and listed in the EPA 537.1, have been analyzed in large water volumes using SPE disks. SDB-XC (Styrene DivinylBenzene) and HLB sorbent were both tested for one liter of water. The analyses were made by LC-MS/MS. Both of the disks demonstrated satisfying results, with slightly better recoveries for HLB disks.

P-47 Optimizing a 190+ Pesticides Multi-Residue Screening Workflow for the Preparation and Analysis of Produce by LC-MS/MS

Landon A. Wiest,¹ Alexandria Pavkovich Bush,¹ Jamie York,² Dan Li,¹ and Joe Konschnik;¹

¹Restek Corporation, 110 Benner Circle; Bellefonte, PA 16823, USA; landon.wiest@restek.com;

²The University of Texas at Arlington, 701 S. Nedderman Drive; Arlington, TX 76019, USA

Pesticides are ubiquitously used to help increase crop yields; however, they can pose risks for public health and pollinators (honeybees). Faster multi-residue screening workflows, which combine easier sample preparation techniques that yield higher recoveries with lower instrument detection limits in fruits and vegetables, are often sought. Accomplishing these goals increases sample throughput, and reduces costs for laboratories and their clients. To demonstrate the feasibility of developing improved methods, organic celery and other representative matrices were spiked with pesticides down to 10 ppb. Samples were extracted using Restek QuEChERS Slim Pouch salts, and cleaned up with complementary dSPE. Each sample was diluted 10x with water prior to analysis. Separations were performed with a Restek Raptor ARC-18 column (100 mm x 2.1 mm, 2.7 μ m) on a Shimadzu Nexera UHPLC. A Shimadzu LCMS-8060 was used for detection. Recovery and precision results from organic celery, spinach and other samples will be shown.

P-48 Quantitation of Mycotoxins in Four Food Matrices Comparing Stable Isotope Dilution Assay (SIDA) with Matrix Matched Calibration Methods by LC-MS/MS

Joe Konschnik, Dan Li, Justin Steimling, Ty Kahler;

Restek Corporation, 110 Benner Circle; Bellefonte, PA 16823, USA joe.konschnik@restek.com

Mycotoxins are secondary fungal metabolites produced by mold that may be found in food or feed. They can cause severe health problems in humans and animals, and can result in significant economic losses. Among the hundreds of toxic mycotoxins, aflatoxins, fumonisins, deoxynivalenol, ochratoxin A, HT-2 toxin, zearalenone, and T-2 toxin are considered as a major concern for corn, wheat, peanuts and other agricultural products. LC-MS has become the standard and is now widely used for routine mycotoxin analysis and identification. One of the challenges faced by LC-MS techniques is the matrix effects caused by the use of electro-spray ionization (ESI). Generally, sample preparation, chromatographic and calibration techniques are the common strategies for reducing the negative effects of matrix effects. Standard addition, matrix matching, and stable isotope dilution assay (SIDA) are all possible calibration solutions. In this work, a quick "dilute-filter-shoot" method was used for sample preparation. A seven-minute LC-MS/MS method using a biphenyl phase column was developed and verified for quantifying twelve mycotoxins in four commodities: corn, peanut butter, brown rice, and corn & wheat mixed. Both SIDA and matrix matched calibration methods were applied, compared, and evaluated in terms of recovery, efficiency, advantages, and limitations.

P-49 Impact of Selected Infrared Wavelengths on Inactivation of Microbes (Fungi and Bacteria) on Rough Rice

Abass Oduola, University Of Arkansas, Fayetteville, AR, USA; aoduola@uark.edu

Rice is one of the most important staple foods in the world. After harvesting, rice is generally dried, and it is susceptible to microbial proliferation especially when stored under inappropriate storage conditions. Hence, it is important to inactivate these microbes by using effective means and technologies. The objective for this study was to investigate the impact of selected infrared wavelength at different time interval followed by tempering step on inactivation of microbes (fungi and bacteria) on rough rice. The sample used for this study was rough rice long-grain (XL 745) with initial moisture content (IMC) of 18.4%. Two-hundred grams (200g) of the sample was treated at different infrared wavelength (λ) such as 3.2, 4.5, and 5.8 μm for 10, 20 and 30 seconds at product-to-emitter gap size of 110, 275, 440 mm. This is then followed by tempering at 60 °C for 4 hours. The fungal count readings were taken 120 hours after plating while the aerobic plate count (APC) readings were taken 48 hours after plating. The samples treated at wavelength 3.2 (product-to-emitter gap 110) for 30 seconds showed the lowest reduction in fungal load and APC; about 3.11 log reduction in the fungal load and about 1.09 log reduction in the APC. On the other hand, the samples treated at wavelength 4.5 and 5.8 shows no significant reduction in the microbial load with respect to the control. However, the tempering stage further reduced the microbial load at each treatment condition. In conclusion, the results show the effect of infrared heat treatment at different wavelengths, time and product-to-emitter gap size, and tempering on reduction of microbes present on the rough rice long-grain (XL 745).

P-50 A Simple, Rapid and Efficient Method for the Extraction of Pesticides from Spices

Candice A.M. Olsson, Brittany Leffler, Benedict Liu, Candice Cashman, and Alicia Stell;

CEM Corporation, 3100 Smith Farm Road; Matthews, NC 28104, USA; candice.olsson@cem.com

Food safety has become a global concern in today's society. Consumers increasingly want to know what is in their food and that the substances they are putting in their body are safe. This, along with stringent regulatory requirements, is leading the call for improved extraction of pesticides from food. The QuEChERS method has been shown to be practical for pesticide analysis in a number of different sample types and is increasingly being employed on more difficult matrices. Unfortunately, some matrices, such as spices, can be challenging with QuEChERS and yield lower recoveries for pesticides than are often observed with matrices that are less complex. While the QuEChERS method is relatively quick compared to other methods, it is also a manual and tedious process. Herein, a rapid, simple and efficient automated method for the extraction of spices showing improved pesticide recoveries is described. The extraction of black pepper, oregano, paprika and cinnamon was performed via extraction on the EDGE and compared to a modified QuEChERS method. The EDGE can utilize the same solvent and clean up sorbents as the QuEChERS method, making it a simple transition for labs currently running QuEChERS. The EDGE extraction allows for extraction, cleanup, and filtration in less than ten minutes. Filtration sub 0.3 μm is possible on the EDGE allowing for direct injection of the extract for UPLC analysis. In this study, the extraction of spices was the focus; however, the EDGE extraction method is applicable to many different types of difficult matrices, such as avocado and cannabis. No matter the matrix, on the EDGE, the same rapid, simple and efficient automated method is sufficient, leading to good pesticide recovery data. The extraction of multiple pesticide residues from spices is described herein, showing improved recoveries and workflow in comparison to QuEChERS.

P-51 The use of DART-HRMS for the identification of BFRs in several food contact polymers and food matrices

R. Paseiro-Cerrato^{1,2}, L. DeJager¹, T. H. Begley¹

¹US FDA, Center for Food Safety and Applied Nutrition, 5001 Campus Drive, College Park, MD, 20740, USA.

²Joint Institute for Food Safety and Applied Nutrition (JIFSAN), University of Maryland, College Park, MD, 20742, USA.

Direct analysis in real time (DART) coupled to a high-resolution mass spectrometer (HRMS) is an ambient mass spectrometric sample introduction technique that can identify a wide variety of chemicals in few minutes. No sample treatment is required to conduct the analysis. DART-HRMS It has been successfully applied in the field of food contact materials (FCM) as well as food as screening technique, obtaining satisfactory results. BFRs are a group of compounds commonly used to prevent flammability of certain industrial products and consequently, to decrease the risk of fire. These types of compounds have been detected in FCM as well as food. The use of BFRs in FCM and therefore as food additives is not allowed in the United States. DART-HRMS was used to rapidly identify BFRs in FCM, food and food simulants. The DART source temperature was 500 °C. The HRMS was set in the negative mode and the mass range was

from 87 m/z to 1300 m/z. Several BFRs standards diluted in toluene and methanol (0.2 – 5 mg/L) were used to validate the method. BFRs were identified in FCA polymers as well as in the food and food simulants. The results suggested that DART-HRMS can be used as rapid screening technique to identify BFRs in the proposed matrices.

P-52 Field Longevity and Attractiveness of Trimedlure Plugs to Male *Ceratitis capitata* in Florida and Hawaii

David Dean¹, Herma Pierre², Lisa Mosser³, Rick Kurashima⁴, and Todd Shelly⁴

¹USDA-APHIS-PPQ-CPHST, 915 10th Street East, Palmetto, Florida 34221, USA;

²NSF Center for Integrated Pest Management, 1730 Varsity Drive, Suite 110, North Carolina State University, Raleigh, NC, 27606, USA; hcpierre@ncsu.edu;

³USDA-APHIS-PPQ-CPHST, 13601 Old Cutler Road, Agricultural Quarantine Inspection Laboratory, Miami, Florida 33158, USA;

⁴USDA-APHIS-PPQ-CPHST, 41-650 Ahiki Street, Waimanalo, Hawaii 96795, USA

Detection of the Mediterranean fruit fly (medfly), *Ceratitis capitata* (Wiedemann) (Diptera: Tephritidae), relies heavily on traps baited with trimedlure, a male-specific attractant. Originally used as a liquid, trimedlure is now dispensed from solid polymeric plugs that reduce volatilization and increase the effective field longevity of the lure. Many of the previous bioassays that measured trimedlure longevity were conducted in Hawaii and adopted a simple “50% rule” for trimedlure plug replacement, i.e., trimedlure plugs should be changed out when their attractiveness (trap captures of male medflies) drops below 50% that observed for fresh trimedlure liquid. The goal of the present study was to assess the field effectiveness of trimedlure plugs in Florida and apply standard statistical analyses to the trapping data. Sterile marked male medflies were released in a citrus orchard with traps containing 3 trimedlure treatments: 2 mL fresh liquid, 2 g fresh plugs, or 2 g plugs aged for intervals of 1, 2, 4, 6, 8, 10, or 12 wk. A single release of approximately 40,000 males was performed for each of the weathering intervals. Weathered trimedlure plugs were as effective as fresh lures when aged 6 wk or less, but catch was significantly reduced for plugs weathered 8 or more wk. At 8 to 12 wk, remaining trimedlure content per plug was ≤ 0.4 g. Ancillary field trials in Hawaii compared male medfly captures in traps baited with fresh 2 or 3 g trimedlure plugs, or 2 or 3 g plugs weathered for 6, 8, or 10 wk. Two and 3 g plugs weathered for 6 wk were as attractive as fresh lures, but 3 g plugs were more effective than 2 g plugs at the longer weathering intervals. Results are compared with prior studies, and implications for medfly management strategies are discussed.

P-53 Determination of Insecticide Residues in Aquacultured Fish Tissue by UHPLC-MS/MS

Ross Potter, Ryan Gibbs, Naomi Harper, Lynn Watson, and Cory Murphy

Canadian Food Inspection Agency, 1992 Agency Drive; Dartmouth, NS, B3B 1Y9, CAN; ross.potter@canada.ca

The Canadian Food Inspection Agency (CFIA) monitors aquacultured finfish for residues of several insecticides used to combat aquatic ectoparasites, such as sea lice. The objective of this research was to make improvements to the current approach by potentially combining three methods into one multi-class/multi-residue extraction method (with one or two detection systems). The result is a method with enhanced efficiency, ruggedness, sensitivity and specificity. All of the compounds of interest share some chemical properties; however, they each have different regulatory requirements. Several are approved for use, and have established Canadian maximum residue limits (MRLs) in salmon (i.e. emamectin, teflubenzuron); others are not permitted for use (i.e. cypermethrin, ivermectin), and the rest are only permitted through Health Canada’s Emergency Drug Release Program (i.e. deltamethrin). The developed method uses a streamlined version of the avermectin and pyrethroid extraction procedures used by the CFIA, with UHPLC-MS/MS detection. Method development and validation has focussed on lipid rich salmonid tissue and has also considered other finfish. The results from method development and preliminary validation data will be presented. Specific emphasis will be placed on challenges and successes encountered with chromatographic separations and matrix induced suppression. It is intended that the current research will enhance the CFIA’s capacity and capability to monitor a variety of aquatic insecticides while providing efficiency gains for the CFIA Dartmouth Laboratory.

P-54 Pilot production of a reference material in fruits with high content of water as a tool for evaluation of laboratories that work in analysis of pesticides residues

Maira A. Paredes¹, Jairo A. Guerrero² and Sergio A. González²

¹Instituto Nacional de Metrología de Colombia, Subdirección de Metrología Química y Biomedicina. Av carrera 50 # 26-55, Bogotá, Colombia; maparedesr@unal.edu.co;

²Laboratorio de análisis de residuos de plaguicidas. Departamento de Química. Universidad Nacional de Colombia. Av carrera 30 # 45-03, Bogotá, Colombia

The reference materials are an important tool to evaluate the quality of the measurement results of a laboratory. For a reference material to be considered suitable for its intended purpose, it is necessary that it be characterized and evaluated with respect to the homogeneity and stability of its properties. In Colombia, few laboratories work in the production of reference materials in pesticide residues, however, currently they do not offer reference materials for this type of analysis. This scenario highlights the need to produce reference materials in the country for this type of tests. In that context, the viability of the pilot production of a reference material for pesticide residue analysis in a purple passion fruit matrix was investigated in this work. The matrix was spiked with 10 pesticides at different levels close to the respective EU MRLs for each analyte selected. Batches of 80 units were produced. The materials' homogeneity (bottle-to-bottle variation), and the mid-term (12 weeks) stability at different temperatures were assessed (-20 °C, 4 °C and 21 °C). For this, an in-house validated LC-MS/MS method with a sample preparation procedure based on the gravimetric QuEChERS principle was applied. The results suggested that the between-unit homogeneity was satisfactory for the intended use, the contribution of the heterogeneity of the samples doesn't exceed 10% uncertainty for any analyte. The storage at -20 °C for 12 weeks did not reveal any detectable material degradation during the evaluated period and their associated uncertainty does not exceed 1%.

P-55 Use of Automated Solid Phase Extraction to Quantify Pesticides in Wastewater in Compliance with EPA Method 608.3

Michael Ebitson and Andrew Taylor

Biotage, 16 Northwestern Dr; Salem, NH 03079, USA; Andrew.Taylor@biotage.com

Many of the pesticides that were used decades ago have now been banned, due to their known impact on the health of animals and humans. To ensure our continued safety, it's important to be able to screen for pesticides in our food and water. The U.S. EPA has published several methods to outline the extraction and quantification of pesticides in water. Some of the methods cover a wide range of contaminants and include pesticide compounds – Methods 525.2, 525.3, 625.1, to name a few. Other methods are a bit more specific to pesticides – Method 549.2 for extracting paraquat and diquat compounds, and Method 608.3 for extracting organochlorine pesticides, for example. The extraction of pesticides can be challenging and processing samples by liquid-liquid extraction can result in poor accuracy and reproducibility. These challenges were taken into account for Method 608 and the latest revision (608.3) was published to formally allow the use of disk-based solid phase extraction (SPE) over liquid-liquid extraction (LLE). In addition to reducing solvent usage and streamlining the extraction process, SPE provides an opportunity to elute your target analytes directly into hexane.

This work will present data to validate the use of an automated solid phase extraction system to extract 21 pesticide compounds from wastewater samples. Target analytes were retained using C18 solid phase extraction media, and the extracts were dried, evaporated and concentrated for quantification by GC-MS. The automated workflow increased sample throughput, while reducing solvent usage and maintaining compliance with EPA Method 608.3.

P-56 Determination of Residual Solvents in Hemp-Based Matrices Using Headspace GC-MS

John Schmitz,¹ Lukas Vaclavik,² Matthew Eckert,¹ Katerina Mastovska¹

¹Eurofins Food Integrity & Innovation, 3301 Kinsman Blvd; Madison, WI 53704, USA; johnschmitz@eurofinsus.com

²Eurofins Food Integrity & Innovation, Otley Road, Harrogate, North Yorkshire, HG3 1PY, United Kingdom

The advent and subsequent proliferation of novel hemp-based consumer products in the last 5-10 years has introduced an abundance of new products to the market. The manufacturers of these products want to use the best extraction method to isolate the desired components from the hemp stalk, seeds, leaves or other parts of the plant. Typical extraction methods use solvents or gases. Residual solvents and gases should be monitored in the raw materials generated or final products produced, in order to evaluate potential contamination of the product. A list of approximately thirty gases and solvents were compiled and evaluated for their performance using a USP <467>-based methodology. Several hemp-based products were spiked with mixes of solvents and gases at low and higher levels in order to establish accuracy and precision of the method. The matrices included ground hemp, ground hemp seeds, hemp oil, CBD (cannabidiol)-infused gummies, and CBD isolate. Additional work was performed to include the many isomers of hexane, heptane, and the common components of petroleum ether. Carbon dioxide was not evaluated, as it is generally regarded as a safe extraction gas, using a supercritical fluid extraction (SFE) methodology. Validation data statistics generated from various matrices at several spiking levels will be presented.

P-57 Maximize the capacity and sensitivity of tandem-mass spectrometry through even distribution of HPLC peaks across the chromatogram

Olusegun Ajayi, Shannon Lane, Irene Cassias, Sarah Wilson, Janet Blount, Darin Files, Andrew Limson, Madison Hanson, Lisa Shin, Eugene Chang*, Kim Thomas-Cruse, [Jacqueline Sram](#)

USFDA Pacific Southwest Food and Feed Laboratory, Irvine, California, USA

High performance liquid chromatography with tandem-mass spectrometry is widely used in chemical residue analysis. The baseline separation in the chromatography is no longer a requirement because of the greater selectivity of tandem-mass spectrometry. However, it does not necessarily mean that there are no limitations regarding co-eluting compounds that cause matrix effects such as ion suppression. Evaluating these limitations of co-eluting compounds using LCMS to ensure the sensitivity, accurate quantitation, robustness and efficiency is important for regulatory work. Based on the dwell time and requirements of data points within a chromatographic peak for quantitation, we defined the number of peaks per minute on a chromatogram as “elution density”. Elution density is used for making a fair comparison between LCMS methods. A good chromatographic method should have fewer co-eluting peaks in a particular period. Such a method distributes the peaks evenly under the gradient ramp of the modifier solvent. Using pesticides compounds, we optimized the LC gradient to distribute the peaks proportionally across the chromatogram. We found that the curved gradient performs better than a linear gradient. This is one of the ways to maximize the capacity and sensitivity in a specific run time of an LCMS or LC-MS/MS method.

P-58 Utilisation of Ion Chromatography and Tandem Mass Spectrometry (IC/MSMS) for the Quantitative Determination of Highly Polar Anionic Pesticide Residues in Fruit and Vegetables.

[Katie Viezens](#), Laura M Melton, Emily E Flynn, Michael J Taylor,

SASA (Scottish Government), Edinburgh, Scotland; Katie.Viezens@sasa.gov.scot

The determination of multiple pesticide residues that could remain in or on our food and drink requires the simultaneous detection, identification and quantitation of hundreds of different pesticides. The variation in chemical and physical properties of individual pesticides and sample matrices combined with ppb residue levels, presents a considerable challenge for analytical chemists.

The group of highly polar anionic pesticides is particularly challenging to analyse since the compounds have poor retention in reversed-phase LC. A robust and sensitive method utilising a hybrid ion chromatography tandem mass spectrometry system (IC-MS/MS) for the simultaneous determination of 9 highly polar anionic pesticides (chlorate, ethephon, fosetyl aluminium, glufosinate, glyphosate, N-acetyl AMPA, N-acetyl glyphosate, perchlorate and phosphonic acid) in fruit and vegetables has been developed to overcome these challenges. Compliance with SANTE/11813/2017 method validation criteria, survey results from the statutory UK/EU Pesticide Residues in Food 2018 programmes i.e. pea, pineapple, melon and successful z-scores for a UK proficiency testing scheme sample (ethephon in pineapple) demonstrate successful application of this IC-MS/MS method. Provision of qualitative determination of AMPA was also facilitated via minor modification of the chromatographic conditions.

P-59 Occurrence of Residues of Veterinary Antibiotics in Water, Sediment and Trout Tissue (*Oncorhynchus Mykiss*) in the South Area of Titicaca Lake – PERU

Franz Zirena Vilca^{1,2,5}, Nestor Cahui Galarza², Juan Rigoberto Tejedo Huaman³, Walter Alejandro Zamalloa Cuba^{2,4}, Clara Nely Campos Quiroz⁵ and Valdemar Luiz Tornisielo⁶

¹ Escuela Profesional de Ingeniería Ambiental de la Universidad Nacional de Moquegua, Urb Ciudad Jardín-Pacocha-Ilo-Perú (franzvilca@usp.br)

² Escuela de Posgrado de la Universidad Nacional del Altiplano Puno Av. Floral 1153 Puno-Perú

³ Regeneration and Cell Therapy Department Andalusian Center for Molecular Biology and Regenerative Medicine – CABIMER. Parque Científico Tecnológico Cartuja Edificio CABIMER Avda Américo Vespucio N° 24 41092 Seville, Spain

⁴ Escuela Profesional de Ingeniería Química de la Universidad Nacional del Altiplano, Av Floral S/N Puno-Perú

⁵ Universidad Nacional Toribio Rodríguez de Mendoza de Amazonas, Calle Higos Urco N° 342-350-356, Calle Universitaria N° 304, Chachapoyas – Amazonas-Perú

⁶ Centro de Energía Nuclear na Agricultura, Laboratorio de Ecotoxicología, Avenida Centenario 303, 13416-903 Piracicaba, SP, Brasil

The production of trout (*Oncorhynchus mykiss*) in Peru has experienced significant growth in the last decade, being the city of Puno the largest producer through intensive systems in floating cages installed in Lake Titicaca. As a consequence it has been described the increase of diseases and the use of antibiotics to control them during the production cycle. In this work we study the impact of antibiotics on drinking water, trout tissues and the lake's aquatic ecosystem. A total of nine veterinary antibiotics were monitored: tetracyclines (chlortetracycline, oxytetracycline and tetracycline), sulfonamides (sulfathiazole, sulfamethazine and sulfadimethoxine) and fluoroquinolones (ciprofloxacin, enrofloxacin, and sarafloxacin). The samples were collected using a non-probabilistic system and analyzed by liquid chromatography coupled to mass spectrometry and solid phase extraction on line (On-line SPE-LC-MS/MS). The sediment samples showed residues of fluoroquinolones (3739.3 µg kg⁻¹) and tetracyclines (3082.9 µg kg⁻¹). Similarly, surface water samples showed concentrations of fluoroquinolones of up to 408.2 and 652.7 ng L⁻¹ in dry and rainy seasons respectively (P>0.05) and samples of drinking water reached an average of 188.1 and 222.2 ng L⁻¹ of ciprofloxacin in dry and rainy seasons respectively. While in trout tissues reached 7.8 µg kg⁻¹ in oxytetracycline 8.7 µg kg⁻¹ in sulfatizole, 4.2 µg kg⁻¹ in ciprofloxacin and 3.6 µg kg⁻¹ in sarafloxacin (P>0.05). The presence of these antibiotics in the trout tissue is attributed to the constant use of these drugs for the prevention and treatment of diseases within the productive system; their presence can have detrimental effects on the aquatic ecosystem, and can even affect public health due to the consumption of aquaculture products and drinking water contaminated with antibiotic residues.

P-60 Depletion of the antibiotic sulfadiazine 14C in rainbow trout (*Oncorhynchus mykiss*)

Franz Zirena Vilca^{1,3}, Olivia M. Luque Vilca², Ricardo Ferraz Silveira⁵, Walter Alejandro Zamalloa Cuba^{3,4} Clara Nely Campos Quiroz⁵ and Valdemar Luiz Tornisielo⁵

¹ Escuela Profesional de Ingeniería Ambiental de la Universidad Nacional de Moquegua, Urb Ciudad Jardín-Pacocha-Ilo-Perú. (franzvilca@usp.br).

² Universidad Nacional de Juliaca, Av. Nueva Zelandia 631, Juliaca – Perú

³ Escuela de Posgrado de la Universidad Nacional del Altiplano Puno Av. Floral 1153 Puno-Perú.

⁴ Escuela Profesional de Ingeniería Química de la Universidad Nacional del Altiplano, Av Floral S/N Puno-Perú.

⁵ Universidad Nacional Toribio Rodríguez de Mendoza de Amazonas, Calle Higos Urco N° 342-350-356, Calle Universitaria N° 304, Chachapoyas – Amazonas-Perú.

⁶ Centro de Energía Nuclear na Agricultura, Laboratorio de Ecotoxicología, Avenida Centenario 303, 13416-903 Piracicaba, SP, Brasil.

Sulfadiazine (SDZ) is an antibiotic belonging to the family of the sulfonamides widely used in animals and humans, reason by which it conducted the study of depletion of this molecule in rainbow trout (*Oncorhynchus mykiss*) with the use of techniques radiometric ¹⁴C with exposure of contaminated feed with sulfadiazine distributed by Sigma-Aldrich track food taking into consideration the weight of fish with a specific activity of 3,5171 MBq mg⁻¹, activity of 9,25 MBq with purity radio chemistry of 98% and chemical purity 95% during 7 days of exposure (absorption - accumulation) and 7 days of purification in semi-static conditions made in the research laboratory of the Professional School of Chemical Engineering of the National University of the Altiplano to their subsequent analysis in the ecotoxicology laboratory of the CENA-University of Sao Paulo - Brazil, where it was the quantification of the antibiotic in the organism (alevino of trout),

water and sediment, conducted from february to June 2018; for which it was using liquid scintillation spectrometry to realize the balance of mass of the applied product. Determined the rate of absorption (k_1), the depuration rate (k_2) and the half-lives biological ($t_{1/2}$) that is 0.65 L kg⁻¹ d⁻¹; 0.16 d⁻¹ and 4.33 d respectively. It was found after the phase of exposure in trout, water and sediment 359,4 ng g⁻¹, 3527,32 ng l⁻¹, and 344,79 ng g⁻¹ respectively and after the depuration phase 131,67 ng g⁻¹, 539,43 ng l⁻¹ and 32,24 ng g⁻¹ respectively.

P-61 Single Class Methodology for Screening and Quantitation of GenX and PFASs in Water and Soil using various Tandem Mass Spectrometry Workflows

Oscar G. Cabrices,¹ Simon Roberts,¹ Craig Butt,¹ Paul Winkler,¹ Scott Krepich² and Christopher Borton;¹

¹SCIEX 1201 Radio Rd; Redwood Shores, CA 94065, USA; oscar.cabrices@sciex.com;

²Phenomenex; Torrance, CA, USA

Due to the environmental persistence of PFOA, PFOS, and other similar compounds, industrial and commercial fluorochemical manufacturers have started producing alternatives. These alternatives have been identified as ether forms of PFOA and other similar compounds. One of these new compounds is referred to as GenX, with the chemical name HFPO-DA. This compound has been detected in the environment at multiple contaminated sites and has been added to the EPA 537.1 method. It is important for any PFAS testing lab to include these compounds in their protocols. In this study, different LC/MS/MS workflows to detect and quantitate HFPO-DA, DONA, and ADONA, in water and soil samples were evaluated. The use of a Scheduled MRM algorithm using a SCIEX QTRAP[®] system that automatically triggers secondary MRM transitions, for increased data quality and confidence in analyte detection at low ng/mL concentration levels is described; as well as the use of a quantitative High Resolution MRM workflow using a SCIEX QTOF system to provide high-resolution, accurate-mass data for full-scan information of both precursor ion and all product ions.

The methodology presented also includes 24 other commonly tested PFAS using two different sample preparation approaches: The first was a dilute-and-shoot method that used an optimized methanol content of 40% to maintain all of the PFAS compounds in solution while maintaining HPLC peak shape. The second approach utilized solid phase extraction (SPE) with ion exchange, mixed mode cartridges, and further cleanup using graphitized carbon. The retention time determined by the optimized reversed phased LC conditions combined with high-resolution mass spectrometry and MS/MS spectra, enabled accurate compound identification across the workflow. On the other hand the sensitivity of the QTRAP System allowed the SPE extract to be injected directly without concentration using nitrogen, which improved recovery of several PFAS compounds, including FOSAs.

P-62 High Resolution, High Sensitivity Analysis of Pesticides in Botanical Dietary Supplements

Jeffrey Dahl and Yuka Fujito

Shimadzu Scientific Instruments, Columbia, MD, USA

Compared with fruits and vegetables, dietary supplements present unique challenges for the detection and quantitation of pesticide residues. The major difficulty is increased levels of non-target substances which interfere and suppress the signal of the target analytes. Even when using highly sensitive and specific techniques such as LC-MS-MS with triple quadrupole mass spectrometry, some false positive signals may be observed from non-target matrix components. Even worse, any substances which are not included in the targeted MRM settings will not be detected.

To overcome these limitations we used a high-sensitivity, high speed quadrupole-time-of-flight mass spectrometer. Using the higher sensitivity and selectivity of this instrument, we developed an LC-MS analysis method using scan and data-dependent MS-MS to detect pesticides in popular dietary supplements. The tandem mass spectra acquired, made possible by the high scan speed, were used to confirm detections.

Representative samples of botanicals were homogenized by grinding and extracted with acetonitrile accompanied by shaking and sonication. Depending on the complexity of the matrix, samples were additionally cleaned up using dispersive solid phase extraction to remove unwanted matrix components. Using our newly developed method, we are able to characterize the extent of residual pesticides present in popular botanicals with unprecedented sensitivity and selectivity.

P-63 Monitoring Tetrachlorvinphos Release from Dog Collars Using Supercritical Fluid Extraction/Chromatography

William Hedgepeth; Yuka Fujita

Shimadzu Scientific Instruments, Inc, Innovation Center, Columbia, MD

Pesticides are added to dog collars to help repel and kill fleas and ticks. These collars are designed to gradually release the pesticides over a period of months to prevent infestation. Recently, there has been growing concern about the pesticide effects on the animal's health as well as humans, especially children, that may come in contact with the pesticides through animal or collar contact. Tetrachlorvinphos (TCVP) is an organophosphate insecticide that works by affecting the central nervous system in fleas, but can also affect human health. TCVP is an EPA listed possible carcinogen, but is still used in dog collars in the US.

On-line supercritical fluid extraction – supercritical chromatography (SFE-SFC) is a new analytical technique for the extraction, separation, and detection of compounds in a single analysis that limits the need for extensive manual sample preparation. The compounds are extracted from a matrix and trapped directly onto an analytical column for chromatographic analysis. This technique was applied to the analysis of the TCVP in store bought dog collars. TCVP values obtained from a store bought dog collar by this technique from package opening to three months will be presented.

P-64 Deuterated Analogues as Internal Standards in the Accuracy of Quantitative Pesticide and Mycotoxin Analysis between Differing Cannabis Matrices

Matthew Texter,¹ and Diana Asensio;²

¹**Shimadzu Scientific Instruments**, 5431 Beaumont Center Boulevard; Tampa, Florida 33634; mgtexter@shimadzu.com; ²Green Scientific Labs Davie, FL

Methods determining the presence and quantitative amount of pesticides and mycotoxins in cannabis products have become important tools in qualifying the safety of patients as regulation for the use of medical marijuana becomes more prevalent throughout the United States. Analysis of these compounds is most accurately performed with the use of a UHPLC system coupled to a triple quadrupole mass spectrometer (LCMSMS). Methods have been created using LCMSMS systems that give accurate and reproducible results when analysis is performed in similar matrices. However, comparisons of these responses in differing complex matrices can lead to changes in quantitative accuracy. These issues are resolved with the use of deuterated analogues as internal standards with a Shimadzu Nexera series UHPLC system coupled to a 8060 triple quadrupole mass spectrometer. Simultaneous analysis of compounds in positive and negative mode, ultra-fast polarity switching, and ultrafast-scanning speeds are shown here to allow for accurate, quantitative, and highly selective analysis. A series of 59 pesticides, 5 mycotoxins and 24 deuterated analogues functioning as internal standards are analyzed in one method with a 15 minute total analysis time. Multiple calibration curves and QC's were prepared and run to demonstrate percent accuracy and RSD values <20%. All calibration curves are linear with R2 values >0.990. These responses were compared between differing matrices to determine the value of deuterated analogues in the accuracy of quantitative results. A variety of matrices were tested using this method and the ability of the deuterated analogues to track quantitative accuracy across matrices is demonstrated.

P-65 Determination of acrylamide in coffee by liquid chromatography-tandem quadrupole mass spectrometry

JD De-Alwis, Euan Ross, Joanne Williams, and Simon Hird

Waters Corporation, Wilmslow, UK; simon_hird@waters.com

Acrylamide is a well-known carcinogenic contaminant formed at high temperatures during the cooking of starch containing foods. Acrylamide hit the headlines again internationally in 2018, when a judge in California ruled

acrylamide fell under the State's Proposition 65 labeling requirements and EU Regulation 2017/2158 was enacted establishing mitigation measures and benchmark levels for the reduction of the presence of acrylamide in food in Europe. The analysis of acrylamide in processed foods has several analytical challenges to consider, including sufficient retention, the complexity of the matrices and the wide range concentrations likely.

A new method, using modified QuEChERS and LC-MS/MS with a high strength silica C18 analytical column, has been developed, to provide a rapid, cost-effective approach for quantifying acrylamide in coffee. Solid phase extraction (SPE) and dispersive solid phase extraction (dSPE) devices were evaluated, to identify simple and efficient cleanup of the samples, to provide selective MRM transitions.

Single laboratory method validation was completed by spiking known amounts of acrylamide into a selection of store purchased coffee products. Acrylamide-d₃ was used as an internal standard to correct for any variability through the whole method, including any LC-MS/MS matrix effects. Validation of the method demonstrated excellent performance in terms of linearity, accuracy, precision and repeatability, in accordance with the criteria outlined in Commission Regulation (EU) 2017/2158. Furthermore, results from the analysis of a coffee reference material demonstrated that the analytical method, using a simple and rapid clean up procedure, was suitable for the determination of acrylamide, in accordance with regulatory requirements.

P-66 Methodology for detection and structural characterization of phosphodiesterase-5 (PDE-5) inhibitor adulterants in an herbal coffee product

Marian Twohig,¹ Andy Aubin,¹ Xiang Jin Song¹, Sarah Dowd,² Gordon Fujimoto,² and Simon Hird³

¹ Waters Corporation, Milford MA, USA;

² Waters Corporation, Beverly MA, USA;

³ **Waters Corporation**, Wilmslow, UK; simon_hird@waters.com

Functional foods require a safe supply of ingredients with verified authenticity. Analysis of coffee products claiming to be a natural solution to erectile dysfunction and stating to be derived from herbs including Tonkat Ali, have been found to contain synthetic PDE-5 inhibitors. The presence of undeclared ingredients can cause risks to health of consumers. There is a need for cost effective monitoring of adulterants in such functional foods.

Here we describe a rapid initial screen using direct analysis in real time (DART) coupled to ACQUITY QDa Mass Detector and confirmation using LC-HRMS. Samples of coffee were analyzed directly, without any sample preparation, using the DART-MS. Ions corresponding to the protonated molecular ions of caffeine, sildenafil and tadalafil at m/z 195, 475 and 390 respectively, were observed in the mass spectra obtained. Analysis of a second coffee sample, generated mass spectra with ions consistent with other PDE-5 inhibitors previously reported in the literature. The same coffee samples were extracted with solvent to perform further studies using untargeted analysis using LC-HRMS. Caffeine, sildenafil and tadalafil were all detected in the extracts by searching the data for entries in a predefined list created from a scientific library and the identify confirmed using accurate mass measurement and isotope patterns of molecular species and fragment ions. Tentative assignments of other suspected PDE-5 inhibitors were made using the Discovery tools available in UNIFI. Isolation of these candidates and further characterization will allow for their definitive identification.

P-67 Determination of legacy and emerging perfluoroalkyl substances in ground and surface waters using LC-MS/MS with direct injection

Kari Organtini, Ken Rosnack, Douglas Stevens, and Jeremy Shia

Waters Corporation, Milford MA, USA; jeremy_shia@waters.com

Perfluoroalkyl substances (PFAS) are common persistent environmental contaminants used in the production of many consumer products as surfactants and for non-stick, stain, and water resistance coatings. PFAS are also a major component of firefighting foams used for suppression of fuel fires. Global use of these compounds over decades has led to their release into the environment and they are found in water, soil, sediment and biota.

Current environmental quality standards, maximum limits and advisory guidelines, set in different parts of the world, require (sub) parts per trillion (ppt) detection of PFAS in various types of environmental samples.

An approach based upon direct injection of water samples was developed for the determination of a wide range of legacy and emerging (e.g. GenX, ADONA, F53-B) PFAS compounds. This approach utilizes little sample preparation and requires a highly sensitive mass spectrometer for detection. Samples of water had been spiked

with a mixture of PFAS compounds prior to being received for analysis. The performance of the method was assessed and passed all the required QC criteria including linearity (R² values > 0.99) and trueness (70 -130%). Detection limits were established to be in the low ppt range. By simplifying the sample preparation step, sample throughput can be drastically increased as well as reducing chances for sample contamination from inherent PFAS in typical laboratory supplies. The performance and scope of the method makes it a suitable approach for the testing of water samples for a wide range of PFAS, legacy and emerging, at relevant concentrations.

P-68 Determination of legacy and emerging perfluoroalkyl substances in ground and surface waters using LC-MS/MS following enrichment by SPE

Kari Organtini, Ken Rosnack, Douglas Stevens, and Jeremy Shia

Waters Corporation, Milford MA, USA; jeremy_shia@waters.com

Perfluoroalkyl substances (PFAS) are common persistent environmental contaminants used in the production of many consumer products. They are used as surfactants and for nonstick, stain, and water resistance coatings and in firefighting foams. Global use of these compounds over decades has led to their release into the environment. PFAS are classified as persistent organic pollutants.

Currently, there are no regulations pertaining to PFAS in water in the USA, although PFOS and PFOA are included in many drinking water health advisories (e.g. 70 ppt [ng/L]). In Europe, the Water Framework Directive and Drinking Water Directive have set minimum quality standards of PFOS and PFOA, which range from the ppb to sub-ppt levels. Such regulations have driven the need for highly sensitive analytical measurements to detect PFAS.

Sample preparation, such as described in the ISO 25101 method, is typically applied for enrichment of PFAS in water samples. The scope of the ISO 25101 procedure has been expanded to cover approximately 40 legacy and emerging PFAS compounds, including GenX, using a weak anion exchange SPE cartridge. The method was assessed using surface, ground, influent and effluent water. The modified method was found to be robust in all types of matrices tested, with detection limits in the low to sub ppt range, making this method suitable for testing compliance with the guidelines/limits set in both the USA and EU. Recoveries were within the prescribed range of 70 - 130 % and method repeatability was assessed with RSDs < 15%.

P-69 Developing a robust LC-MS/MS method for the determination of anionic polar pesticides in a range of foodstuffs without derivatization

Dimple Shah¹, Benjamin Wuyts², Euan Ross², Simon Hird³, Keil Brinster¹

¹**Waters Corporation**, Milford, USA,; ²Waters Corporation, Belgium, ³Waters Corporation, UK

Glyphosate continues to cause controversy and so analysis is of considerable interest to governments, the food industry and contract testing laboratories. Many wish to move away from methodology that employs derivatization to save time and expand the scope to cover other polar and ionic pesticides.

Chromatographic retention and separation was optimized using a novel hydrophilic interaction liquid chromatography (HILIC) column, applying an acidified mobile phase gradient, with and without ammonium formate. The performance of a buffered and un-buffered version of the method was compared. Removal of the ammonium formate from the mobile phase resulted in improved sensitivity without compromising chromatographic performance. The aim was to achieve chromatographic retention and baseline separation of isobaric compounds whilst providing maximum sensitivity of all target analytes.

Foods of plant origin were prepared using a modified version of the Quick Polar Pesticides (QuPPE) extraction procedure and spiked with a panel of representative anionic polar pesticides for analysis. All analytes were sufficiently retained detected at concentrations <0.01 mg/kg in matrix-matched standards using the new acidified method. All isobaric pairs (AMPA/fosetyl al and fosetyl al/phosphonic acid) were well separated. The performance of the LC-MS/MS method was assessed using the relevant criteria defined in the SANTE guidance document (SANTE/11813/2017). Linearity was assessed through matrix-matched calibration over a suitable concentration range (0.001-0.1 mg/kg). Ion ratios and retention times agreed well with reference values and all were within the required tolerances ($\pm 30\%$ and ± 0.1 minutes, respectively). The details of a new improved LC-MS/MS method will be presented.

P-70 Determination of Pesticides in Edible Oils

Dimple Shah¹, Michael Young¹, Euan Ross², Janitha De-Alwis² and Stuart Adams²

¹Waters Corporation, USA, ²Waters Corporation, UK,

Detection of pesticides in olive oil has many challenges due to the complexity of the matrix which is high in lipids. Sample cleanup is, therefore, crucial to ensure robust methodology. Traditional method involved liquid-liquid extraction followed by gel permeation chromatography (GPC) cleanup. This technique is undesirable due to high solvent consumption and lengthy extraction and cleanup times. This poster presents an alternative, extraction and cleanup procedure optimized for the simple and reliable determination of multi-residue pesticides in edible oils by GC-MS/MS.

Edible oils were homogenized and spiked with known concentrations of pesticides. The extraction was optimized, followed by a simple pass through cleanup. All extracts were run on GC-EI-MS/MS, in MRM mode, using a splitless injection of 1 μ L. Validation was completed in accordance with European guidelines (SANTE/11813/2017) evaluating sensitivity, selectivity, repeatability, accuracy and identification criteria.

The extraction optimized in this study yielded improved method recovery for representative pesticides, when compared to the traditional hexane and acetonitrile extraction procedures. The impact of the pass through cleanup step was also evaluated separately, where analyte recoveries were >70% for all analytes. An alternative type of calibration, namely procedural standards, was employed. This mode of calibration compensates for low extraction efficiency and matrix effects and showed excellent improvements in terms of accuracy and repeatability, where the method's trueness ranged from 99.2 to 108.5 % for a selection of challenging organochlorine pesticides.

Excellent linearity, over the calibration range of 0.005 to 0.1 mg/kg, was achieved for all pesticides with coefficients of determination > 0.995 with residuals <20%. The method's accuracy, repeatability and bias was evaluated from the analysis of spikes at 0.01 mg/kg (n=5), 0.02 mg/kg (n=5) and 0.1 mg/kg (n=5).

P-71 Optimization of Pass-Through SPE Cleanup for LC-MS/MS Multi-Residue Veterinary Drug Analysis

Michael S. Young, Melvin Blaze, and Kim Tran

Waters Corporation, 34 Maple Street, Milford MA

Before the introduction of the highly sensitive and selective LC-MS instrumentation commonly used for today's multi-residue methods, compound or class specific analytical methods were required to meet regulatory requirements. These methods often involved cumbersome and time-consuming multi-step analyte isolation and enrichment followed by cumbersome and time-consuming multi-step cleanup. Today's residue methods require much less analytical rigor. A simple extraction procedure followed by a simple cleanup step (such as pass-through SPE) may be suitable for accurate determination of hundreds of analytes from complex matrices. Although simple, such extraction and cleanup methods are not fool-proof; variables in the extracts, such as water content, protein content, pigment content, and lipid content, need to be considered to maximize cleanup and avoid recovery losses of individual compounds or compound classes. In this presentation, multi-residue veterinary drug analysis will be discussed with emphasis on the optimization of a pass-through SPE cleanup protocol.

P-72 Application of GC-EI-TOF-MS using Large Volume Injection for pesticide residue analysis in fruit and vegetables

Maria del Mar Gomez-Ramos, Maria Murcia-Morales, Amadeo R. Fernández-Alba

University of Almería, Department of Physics and Chemistry, Agrifood Campus of International Excellence (ceiA3), Ctra. Sacramento s/n, La Cañada de San Urbano, 04120, Almería, Spain

Pesticide residue analysis on fruit and vegetables is essential for the protection of human health and to guarantee

international trade. The great number of pesticide residues and the wide range of concentration encountered means that it is necessary to use powerful analytical tools capable of identifying and quantifying a high number of residues at trace levels. Nowadays, the analysis of pesticides is dominated by triple quadrupole (QqQ) technology. However, the recent advances of high-resolution-accurate-mass-spectrometry (HRAMS) instruments make its performance in quantitative analysis fully adequate. This study shows the feasibility of GC-TOF-MS using large volume injection for the multiresidue analysis of 214 pesticides in QuEChERS extracts of tomato and orange. A high degree of sensitivity was achieved with an instrumental limit of identification (LOI) of 2 µg kg⁻¹ for the 86 % of the compounds in tomato and 75% in orange matrix. The instrument presented very good linearity over the concentration range 5-100 µg kg⁻¹ ($r^2 > 0.99$). Robustness of analytical conditions was measured thorough 40 consecutive injections in orange matrix showing good response, with the intensity of the signal not affected in 75 % of the pesticides. Very good mass accuracy was observed with values < 2 ppm for most pesticides in tomato and orange. The instrument showed great capabilities for the identification and quantification for pesticide residues in fruit and vegetables in agreement with EU AQC procedures. In addition, the high selectivity provided by the instrument allowed the detection of false positives and negatives identified by QqQ.

P-73 Analysis of pesticide residues in dried spices by supercritical fluid chromatography coupled to tandem mass spectrometry

Victor Cutillas, María Murcia-Morales, María del Mar Gómez-Ramos, Amadeo Rodríguez Fernández-Alba

European Union Reference Laboratory for Pesticide Residues in Fruit and Vegetables; Agrifood Campus of International Excellence (CeIA3), Department of Chemistry and Physics, University of Almería, 04120 Almería, Spain; amadeo@ual.es

Spices are complex matrices that contain large amounts of essential oils, plant nutrients and secondary metabolites such as flavonoids, terpenes and alkaloids. These interfering matrix components produce ion enhancement or suppression which can be very strong and depend on the origin of the sample. This study describes the improvement of sensitivity and the reduction of the ion suppression that can be achieved by supercritical fluid chromatography (SFC) in the analysis of dried spices as difficult matrices. Black pepper and cayenne were used for the validation of 162 pesticides. The validation study was performed in terms of recovery, linearity, matrix effect, intra-day and inter-day precision. Different clean-up sorbents were tested for both matrices: PSA, Z-Sep and EMR. At least 95% of the compounds showed good recoveries for both matrices at the selected concentration levels (50 µg kg⁻¹ and 200 µg kg⁻¹). Regarding matrix effect, only 6% of the compounds in cayenne and 17% in black pepper showed strong signal suppression. Good results were also obtained in terms of precision and linearity. Most of the pesticides studied met the requirements to be identified at the lowest concentration level of 5 µg kg⁻¹ in both matrices. 47 real samples of different types of species and different origins were analyzed using the validated method. 81% of the samples presented one or more pesticides and 46 different pesticides were identified. Despite the complexity of the matrix, the results obtained in the validation showed that supercritical fluid chromatography facilitates the analysis in these complex matrices.

P-74 Separation of chiral pesticides by applying supercritical fluid chromatography coupled to tandem mass spectrometry

Victor Cutillas and Amadeo Rodríguez Fernández-Alba

European Union Reference Laboratory for Pesticide Residues in Fruit and Vegetables; Agrifood Campus of International Excellence (CeIA3), Department of Chemistry and Physics, University of Almería, 04120 Almería, Spain; amadeo@ual.es

Supercritical fluid chromatography (SFC) is a well-known technic used for enantioseparation in the pharmaceutical industry but its application in pesticides field has not been well studied yet. In this work 21 pesticides were separated using SFC-MS/MS with a polysaccharide-based chiral stationary phase column. Supercritical fluid chromatography allows the separation of isomers in a short run time because high flow rates can be applied without losing chromatographic efficiency. A change of the mobile phase used for the multiresidue method is not necessary, in addition, the absence of water and the low flow that reaches the source

increase the sampling efficiency. This research focuses on those compounds whose isomers have a different toxicological nature like Lambda-Cyhalothrin. Lambda-Cyhalothrin is a mixture 1:1 of lambda and gamma isomers. The acute reference dose (ARfD) of gamma isomer is twice that of lambda. Lambda-Cyhalothrin was applied in lettuces under greenhouse conditions. Collections were made at different stages and analyzed to identify if there is any change in the isomers proportion. A validation study of lambda and gamma cyhalothrin was performed in terms of linearity, matrix effect and precision. The limit of quantification (LOQ) was 5 µg/kg-1 in the different matrices studied. In conclusion, the results obtained in the validation showed that SFC-MS/MS facilitates the enantioseparation of pesticides within a short period of time and obtaining very good LOQs of each isomer.

P-75 Development of a method for the decontamination of pesticides in beeswax foundation

Antonio Rodríguez Fernández-Alba², Raúl Alonso del Águila², M.J. Gomez-Ramos¹, Victor Cutillas¹, M.D. Hernando³, Amadeo Rodríguez Fernández-Alba¹;

¹**European Union Reference Laboratory for Pesticide Residues in Fruit and Vegetables**; Agrifood Campus of International Excellence (CeIA3), Department of Chemistry and Physics, University of Almería, 04120 Almería, Spain; amadeo@ual.es

²Department of Chemical Engineering, University of Alcala, Alcala de Henares, 28801 Madrid, Spain;

³National Institute for Agricultural and Food Research and Technology - INIA, 28040 Madrid, Spain;

Beeswax foundations are commonly used in beekeeping to facilitate the construction of the honeycomb by bees. Beekeepers apply acaricides on the wax to improve the fight against varroa destructor mite. Some of these pesticides, such as Coumaphos, Chlorfenvinphos, and Fluvalinate-Tau are lipophilic being absorbed into the wax. These beeswax foundations are usually recycled but pesticides remain, and they accumulate during each fumigation. In this study, decontamination methods were applied using different parameters: quantity of solvent added per amount of matrix, type of solvent used (methanol, water, mixture), temperature conditions, waiting time, type of reactor where the experiment takes place and pressure applied inside it. The beeswaxes were analyzed before and after the decontamination method using gas chromatography and supercritical fluid chromatography both coupled to mass spectrometry. Multiresidue methods were applied to identify other pesticides present in these samples. Aside from the already mentioned acaricides, 12 pesticides were detected in beeswaxes at low concentrations being Acrinathrin the pesticide most detected after the decontamination. Out of the 51 decontamination procedures applied, 5 produce a reduction higher than 90% of the pesticide load for Coumaphos and Chlorfenvinphos, being 4 in the case of Fluvalinate-Tau. The most efficient method reduces acaricides concentration by 98%. In conclusion, decontamination methods developed showed to be efficient in removing the pesticides from the beeswaxes and avoiding acaricides accumulation.

P-76 Evaluation of influence of the number of mass windows in QOrbitrap (vDIA MS2) on detection and identification of pesticides

Łukasz Rajski, Iciar Beraza Gomez, María José Gómez Ramos, Amadeo R. Fernández-Alba

University of Almería, Department of Chemistry and Physics, Agrifood Campus of International Excellence (CeIA3), **European Union Reference Laboratory for Pesticide Residues in Fruits and Vegetables**, Ctra. Sacramento s/n La Cañada de San Urbano, 04120 Almería, Spain; amadeo@ual.es

A grate advantage of high-resolution mass spectrometry is that in this technique the extensive information about analyzed sample can be obtained. It is possible because the instrumentation offers fast and sensitive scanning modes. A non-targeted data independent acquisition is possible in MS as well as in MS2 mode. A mass range of m/z 100 – 950 includes majority of LC amenable pesticides. In MS this range is acquired in one scan. Before the subsequent scan the ions from mentioned range can be sent to the collision cell, fragmented and all product ions are also acquired in one scan. However, in MS2 the mass range of interest can be divided by the quadrupole into smaller subranges which are fragmented separately and registered in the separated scans. In theory this approach helps to reduce the number of interfering ions and some cases increase the sensitivity of MS2 fragment

ions. In this study a QExactive Focus (QOrbitrap) mass spectrometer was used. Parent ions from the m/z 100 – m/z 950 were fragmented in one, five, and eight fragmentation events. Two approaches were tested here- fixed and variable mass window width. A group of 162 pesticides in three vegetal matrices (tomato, orange, leek) and in pure solvent was evaluated. In case of fixed window size, the lowest number of detected MS₂ ions was obtained for 5 mass windows. Method with 8 mass windows showed slightly better results than the method with 1. In comparison to the fixed, the variable window size improved the results.

P-77 Identification and evaluation of chemical residues in honeybee larvae

María Murcia Morales, María José Gómez Ramos, Francisco José Díaz Galiano, M^a del Mar García Valverde, A. R. Fernández-Alba

Agrifood Campus of International Excellence (ceiA3), Department of Chemistry and Physics, University of Almeria, European Union Reference Laboratory for Pesticide Residues in Fruit & Vegetables, 04120 Almería, Spain, amadeo@ual.es

The increasing use of pesticides in agricultural and apicultural practices is a factor contributing to the reduction of honeybee populations. Bees are subjected to unintentional exposure to pesticides applied to crops during their foraging activities. However, the highest pesticide residues found in honeybee colonies are acaricides that have been intentionally introduced into their hives in an attempt to mitigate the effects of Varroa and other infestations. Honeybees may also be exposed to chemical compounds during larval development by contact with the beeswax or orally through the bee bread. Despite the larvae's intense food intake, it does not eliminate the excreta until the end of the larval development, bioaccumulating the xenobiotics until the beginning of its metamorphosis. Most studies into pesticide exposure assessment on honeybees are focused on adult bees, even though bee brood (immature bees: eggs, larvae and pupae) is crucial to colony welfare. The present study evaluates the presence and distribution of chemical residues in beeswax, bee bread and honey and their migration to bee brood and determinates the extent of the larvae exposure. Samples were collected from an experimental apiary, managed according to a conventional production, during six months. Their analysis by LC-MS/MS and GC-MS/MS with wide scope multiresidue methods showed residues of acaricides -coumaphos, fluvalinate-tau, acrinathrin and cypermethrin- in the larvae and at higher levels in wax and bee breed. These results show the accumulation process of residues in beeswax and the diffusion into the beehive environment, including the bee brood.

P-78 Evaluation of SWATH[®] acquisition mode in LC-ESI-QTOF-MS for the identification and quantification of pesticide residues in complex matrices

Iciar Beraza Gómex, María José Gómez Ramos, Łukasz Rajski, A. R. Fernández-Alba

Agrifood Campus of International Excellence (ceiA3), Department of Chemistry and Physics, University of Almeria, **European Union Reference Laboratory for Pesticide Residues in Fruit & Vegetables**, 04120 Almería, Spain; amadeo@ual.es.

The introduction of data independent acquisition by the sequential window acquisition of all theoretical fragment ions (SWATH[®]) in high-resolution quadrupole time-of-flight analysers undoubtedly represents an important improvement in the MS/MS spectra obtained when working in non-target analysis. However, the advantages and limitations of this approach have not been sufficiently evaluated. This study evaluate SWATH[®] method, which combines MS and MS/MS acquisition, dividing the entire mass range into smaller segments for the MS/MS mode. The effect of the number of mass isolation windows, the total cycle-time lapsed, the sensitivity obtained, the MS/MS spectra quality, the ion ratio stability as well as identification and quantification capabilities have been evaluated. The study was carried out on 163 pesticides in tomato, leek and orange matrices at different concentrations (1, 5 and 20 ppb) and using different number of mass isolation windows for data acquisition (1, 5, 8 and 20 mass isolation windows). Special attention was given to certain issues that can make correct identification difficult, such as matrix influence in different areas of the chromatogram, the effect of concentration, the mass window width, and automatic identification with a library. The reasons why some of the compounds were not identified were because they did not fulfil the ion ratio criterion, the compound was

not detected at all or one of the ions had a mass error above 1 mDa. In general, narrowing the mass isolation window improves the selectivity and the sensitivity in MS/MS mode because less matrix interferences arrive at the collision cell at the same time as the analytes.

P-79 Considerations for automation of PFAS analysis using EPA method 537.1

Haibin Wan, **PromoChrom Technologies Ltd.**, Unit 6, 12180 Horseshoe Way, Richmond, BC V7A 4V5, Canada.
Wanhaibin6@hotmail.com

Sample extraction for analysis of PFAS in water using a vacuum manifold is labor intensive and time consuming due to complex procedures and difficulties in controlling the flow rate. There is a high demand among the testing laboratories for automation of the solid phase extraction procedures. The process of automation is much more challenging than in case of other SPE methods. This is mainly because the EPA method specially requires that changes may not be made to sample extraction steps. To make the work of automation more difficult, the very low reporting level requires a very clean background from the extraction equipment. Many commonly used Teflon parts need to be replaced with parts of other inert materials. Since July 2018, we have been working with customers from the United States and Australia to automate the sample extraction procedures for PFAS analysis. The work has resulted in a well accepted automation solution that is fully automated, fast, clean, and robust. This presentation will share the experience in instrumentation and in transfer of vacuum manifold based method to an automated method. Factors affecting the recovery and background level will be discussed.

P-80 Applying High-Resolution GC-Orbitrap Mass Spectrometry to Quantitation of Pesticides and PCBs in Orange and Pepper Extracts

Jason Cole¹, Dominic Roberts², Jim Garvey³, Richard Law² and Cristian Cojocariu²

¹ **Thermo Fisher Scientific**, Austin, Texas, USA; jason.cole@thermofisher.com

² Thermo Fisher Scientific, Tudor Road, Manor Park, Runcorn, United Kingdom; dominic.roberts@thermofisher.com; richard.law@thermofisher.com; cristian.cojocariu@thermofisher.com

³ Department of Agriculture, Food and the Marine, County Kildare, Ireland; jim.garvey@agriculture.gov.ie

In this study, gas chromatography (GC) coupled to high resolution Orbitrap-based mass spectrometry (HRMS) was used for quantitative assessment of pesticide residues and polychlorinated biphenyls (PCBs) in orange and pepper samples. The default acquisition mode was full scan accurate mass (untargeted) allowing all the ions to be acquired at the same time across a specified mass range, simplifying instrument operation and method setup and giving the analyst the flexibility to decide post-acquisition which pesticides and ions to measure. The high resolution capability in combination with low limits of detection increases the scope of the analysis without the need for optimization of individual compound acquisition parameters. The first objective of the study was to assess the linearity of response over a calibration range of 1-200 ppb ($\mu\text{g}/\text{kg}$). The linear response as indicated by the coefficient of determination (R^2) was >0.994 and the residual average response factor RSD% was $< 20\%$ for all compounds. Secondly, the system sensitivity was tested by repeat injections of serially diluted matrix-matched orange and pepper samples spiked with up to 25 compounds at various levels, establishing the limit of detection (LOD). To assess the accuracy of quantitation, an orange and pepper sample were analyzed blindly after being post-spiked with compounds at concentrations varying from 0.5-120 $\mu\text{g}/\text{kg}$. Experiments to assess the stability of mass accuracy and stability of the response were repeated in two different days for each matrix.

P-81 UHPLC-MS/MS analysis of neonicotinoids and their metabolites in plant tissues and pollen by modified QuEChERS

Viet Dang¹, Dave Borts¹, Maura J. Hall², Ed George³

¹Veterinary Diagnostic Laboratory, Department of Veterinary Diagnostic and Production Animal Medicine
College of Veterinary Medicine, Iowa State University, Ames, IA USA

²Department of Entomology, Iowa State University, Ames, IA USA

³Thermo Fisher Scientific, LSMS Food and Beverage CSC, San Jose, CA USA

Neonicotinoids are a relatively new class of insecticides extensively used in seed and soil treatment. They act as nicotinic acetylcholine receptor (nAChR) agonists, which affect the central nervous systems of insects resulting in paralysis and death. They can be taken up through the roots of plants and translocated to their leaves, flowers, and pollen. Up to date, it is still challenging to have a comprehensive LC-MS workflow for the simultaneous measurements of imidacloprid, thiamethoxam, clothianidin, and imidacloprid metabolites (e.g., OH-imidacloprid and imidacloprid olefin).

A generic QuEChERS extraction was applied for pollen and plant material. Samples were spiked with labeled internal standards, extracted with acetonitrile, and salted out with MgSO₄ and NaCl. The mixture was centrifuged, and 1 ml of supernatant was subjected to dispersive solid-phase extraction (dSPE) clean-up. Analysis of neonicotinoids and metabolites was performed on a UHPLC with tandem triple quadrupole (LC-MS/MS). LC separation was carried out on a C18 column (100 × 2.1 mm, 2.6 μm) with injection volume of 2 μl. The mobile phases consisted of A: water:methanol (98:2 v/v) with 5mM ammonium formate and 0.1% formic acid and B: methanol:water (98:2 v/v) with 5mM ammonium formate and 0.1% formic acid.

Results showed good linearity over the concentration range for neonicotinoids and metabolites and correlation coefficients were at $r^2 > 0.990$. Method detection limits (MDLs) ranged from 0.05 – 0.3 ng/g in plant tissue, and from 0.1 – 1 ng/g in pollen. The intra- and inter-day precision (%RSD) were all within 15% of the reference values, and the accuracy ranged from 78 to 110%. Application of dSPE for sample cleanup indicated minor matrix effects when comparing the peak response of analytes in post-spiked extracts with the peak response of analytes in pure solvent. The extraction recoveries ranged from 85 to 101% for target analytes in plant tissues and pollen.

P-82 Evaluation of a comprehensive multi-class veterinary drug analytical method using a certified reference material of drug residues in bovine muscle

Ed George¹, Dipankar Ghosh¹, David Borts², Viet Dang²

¹Thermo Fisher Scientific, LSMS Environmental and Food Safety, San Jose, CA USA

²Veterinary Diagnostic Laboratory, Department of Veterinary Diagnostic and Production Animal Medicine College of Veterinary Medicine, Iowa State University, Ames, IA USA

Veterinary drugs are broadly defined as chemicals that are used to protect animals from contracting disease, promote growth, and in some cases provide aesthetic qualities in food production. The inappropriate use of veterinary drugs can have adverse effects on animals, the environment, and human health.

The determination and efficient analysis of veterinary drugs is an important part of routine food quality control.

The European Union (EU) and others have developed specific regulations to address these growing concerns.

The requirements of low limits of quantification in diverse matrices, along with a wide variety of chemical classes and properties of veterinary drugs pose significant analytical challenges. Several methodologies exist which are typically limited in scope to specific chemical classes, labor intensive, and require extensive sample preparation and clean-up.

This study applies a multi-residue, multi-class workflow using a simple modified QuEChERS sample preparation procedure to the analysis of over 160 veterinary drugs by liquid chromatography-triple stage mass spectrometry (LC-MS/MS). The workflow will be evaluated using a certified reference material known as BOTS-1. It contains incurred veterinary drug residues in bovine muscle. The certified values are based on results from the National Research Council Canada (NRC), the Canadian Food Inspection Agency (CFIA), the USDA, and the German Federal Office of Consumer Protection and Food Safety (BVL) using tandem LC-MS/MS. The sample preparation method and analysis of the BOTS-1 sample will be described, along with performance data obtained vs. the certified values.

P-83 New developments in IC-MS/MS for the multi-residue analysis of pesticides including polar anionic pesticides and metabolites in food

Ed George¹, Richard Fussell², Fausto Pigozzo³, Tao Bo⁴, Qilei Guo⁴, Yingchen Li⁴

¹Thermo Fisher Scientific, LSMS Food and Beverage CSC, San Jose, CA USA

²Thermo Fisher Scientific, Milan, Italy

³Thermo Fisher Scientific, Hemel Hempstead GB

⁴Thermo Fisher Scientific, Food and Beverage China CSC, Beijing, China

Laboratories are constantly challenged to analyze more classes of pesticides at lower concentrations in several different commodities, with faster turnaround times and little if any increase in costs. They will also expect that residues will not go undetected and all results can be verified by associated analytical quality control data compliant with specific method performance guideline criteria.

One of the most challenging groups of pesticides are the polar anionic pesticides, such as glyphosate, perchlorate, chlorate and the like, which often occur as residues in food, but are not always included in pesticide monitoring programs. A number of analytical approaches were considered, including HILIC, and non-suppressed IC, but IC with eluent suppression offered a number of benefits. This presentation will therefore focus on the development of a new, validated IC-MS/MS based workflow for the robust, sensitive and reliable determination of 15 polar anionic pesticides and metabolites at low µg/kg levels in a single run. The development was not straightforward so the presentation will highlight the challenges encountered and steps taken to successfully overcome these.

The workflow uses a modified Quick Polar Pesticide (QuPPE) extraction with cartridge solid phase extraction clean-up and has been thoroughly tested and validated. Results for wheat and leek matrices are compliant with SANTE guidelines, and EU MRLs. Quantification limits are 10µg/kg with % RSDs typically <10 %. Recoveries with and without internal standards and using matrix-matched calibration, and procedural matrix extracted calibrations will be presented.

P-84 A new single, multi-analyte, robust and sensitive “sample-to-result” IC-MS/MS Workflow for the analysis of polar anionic pesticides and contaminants

Qilei Guo,¹ Yingchen Li,¹ Fausto Pigozzo,² Richard Fussell,³ and Tao Bo;¹

¹ **Thermo Fisher Scientific**, Qiming Plaza, Chaoyang District, Beijing; 100102, China; qilei.guo@thermofisher.com; ² Thermo Fisher Scientific, Strada Rivoltana, Milan, Italy; ³ Thermo Fisher Scientific, Stafford House, Hemel Hempstead, UK

Polar pesticides include some of the most frequently used pesticides such as glyphosate and glufosinate. Due to the complexity of food matrices and the high polarity of anionic pesticides, their simultaneous and sensitive analysis is challenging. This presentation provides information on the development and validation of a new integrated ‘sample-to-results’ workflow for the reliable and sensitive quantitation of polar anionic pesticides and contaminants in food. The workflow is based on the use of high capacity ion exchange columns with post column eluent suppression coupled to a high sensitivity triple quadrupole mass spectrometer (IC-MS/MS).

The workflow is based on a modified version of QuPPE (Quick Polar Pesticides) extraction procedure. In QuPPE method, the absence of a liquid partitioning step and/or solid phase clean-up step, results in ‘dirty extracts’ containing high concentrations of matrix co-extractives, thus the separation and accurate quantification of analytes in QuPPE extracts is difficult. Analysts attempt to mitigate these issues by the use of labelled internal standards and various SPE cartridges.

The results demonstrate that the workflow based on IC-MS/MS with clean-up step can overcome many of the issues experienced with previous methods reported for the analysis of polar pesticides. the IC-MS/MS workflow approach enables aggregation of separate methods into a single analysis improving productivity, while the high capacity ion exchange columns can withstand higher sample loading enabling the analysis of lower concentrations of polar analytes in the more difficult, but relevant matrices, such as cereals and cereal products.

P-85 A Multiresidue Method for Quantitation and Screening of Pesticide Residues in Baby Food Using LC-MS/MS

Anastasia Kalli, Charles Yang, Ed George and Dipankar Ghosh

Thermo Fisher Scientific, San Jose, CA, USA

A large number of pesticide residues is used worldwide in food products for preventing, destroying or controlling pest activity. Therefore, for consumer protection, regulatory agencies have established maximum residue levels

(MRLs). For baby food products MRLs are even lower compared to other food commodities, and therefore sensitive analytical methods that allow simultaneous analysis of a large number of pesticides in challenging matrices are required. We have developed a 15-min multiresidue method for quantitation and screening of pesticide in baby food using a triple quadrupole mass spectrometer, coupled to a HPLC in a single run with polarity switching.

Baby food was obtained from a local retail store. Samples were extracted using a QuEChERS extraction kit. Briefly 10g of sample was weighed and 10ml of ACN was added. The mixture was shaken followed by addition of salts from a pre-prepared QuEChERS extraction mix and then shaken again. Followed centrifugation the supernatant was collected. Pesticide standards were spiked into the matrix extracts at different concentration levels ranging from 0.05 to 200 ppb. Chromatographic separation was performed on a Vanquish UHPLC system using an Accucore aQ column. Mass spectrometric analysis was performed on a TSQ Quantis triple quadrupole mass spectrometer with polarity switching. Data analysis was performed with Trace Finder software.

We have developed a multiresidue LC-MS/MS method for the analysis of pesticides in baby food using a triple quadrupole mass spectrometer. The 15-min method allows for pesticide quantitation and screening at low concentration levels (ppb) which are required for baby food. Pesticide confirmation was performed based on one or two ion ratios. Optimum SRM transitions (quantifier and qualifier ions) were determined for each compound by optimizing RF lens values and collision energies for each of the 250 pesticides in neat standards. Preliminary data obtained from matrix-matched standards indicates that the performance of the method in terms of RT reproducibility, detection limits, CVs, RSDs, accuracy is excellent. Limit of detection and limit of quantitation were between 0.5 ppb to 5 ppb for all pesticides tested. Future work will focus on method validation according to the SANTE guidelines. We will discuss in details the data, results and method validation.

P-86 A Multiresidue Method for Pesticide Analysis Using an Orbitrap Tribrid Mass Spectrometer and Automatic Background Exclusion

Anastasia Kalli, Seema Sharma, and Dipankar Ghosh

Thermo Fisher Scientific, San Jose, CA, USA

Pesticides are routinely applied to crops for preventing, destroying or controlling pest activity. Given the large number of pesticides used and the globalization of the food industry, multiresidue methods offer a great advantage allowing analysis of hundreds of pesticides in a single run. We have implemented a multiresidue method for the analysis of 250 pesticides on an Orbitrap ID-X Tribrid mass spectrometer utilizing an automatic background extraction workflow (AcquireX).

Strawberry samples were obtained from a local retail store. Following homogenization, strawberry samples were extracted using a QuEChERS extraction kit. The matrix extracts were spiked with the pesticide standards (250 pesticides) at different concentration levels ranging from 0.05 to 200 ppb. Chromatographic separation was performed on a Vanquish UHPLC system using an Accucore aQ column. Mass spectrometric analysis was performed on an Orbitrap ID-X Tribrid mass spectrometer using AcquireX workflow, for automated generation of background exclusion list, or data dependent acquisition (DDA).

We have evaluated the performance of a multi-residue pesticide method utilizing high mass accuracy and high resolution for semi-quantitation and screening of pesticide residues in a strawberry matrix. Excellent detection limits, reproducibility, linearity and accuracies were obtained. Overall, for 250 pesticides, out of 251 tested, the LODs were at/or below 5 ppb with 215 pesticides having LODs at/or below 1 ppb. LOQs were below 5 ppb for 247 pesticides tested. When the AcquireX workflow was applied for automated background exclusion we observed a significant increase in the number of library matches compared to DDA, especially at the lower concentration levels. For instance, at a spiked concentration of 0.5 ppb the presence of 19 pesticides was confirmed via library search with DDA. When utilizing the AcquireX workflow, at the same concentration level, the presence of 145 pesticides was confirmed. Similar trends were observed at a concentration level of 1 ppb in which we observed 178 library matches with AcquireX versus 65 library matches with DDA.

P-87 Fast, ultra-sensitive analysis of PBDEs in food using advanced electron ionization GC-MS/MS technology

Aaron Lamb¹, Sean Panton², Adam Ladak³, Carlos Garcia³, Tim Anderson³, Paul Silcock¹, Cristian Cojocariu¹

¹Thermo Fisher Scientific, Runcorn, UK

²Fera Science Ltd, York, UK

³Thermo Fisher Scientific, Austin, TX, USA

PBDEs are additive flame retardants used in consumer goods, the major problem is that these compounds leach into the environment where they persist and bioaccumulate and are toxic at very low concentrations. Due to recent innovations in triple quadrupole GC-MS/MS technology lower levels of detection are now achievable for PBDEs in food. The experiments described focus on quantitative performance assessment of triple quadrupole GC-MS/MS technology. Data were acquired using timed-SRM mode which provides high selectivity and sensitivity for targeted PBDE analysis. All target congeners were separated in <11 min with excellent separation of the critical pairs (ex: BDE-49 and BDE-71 with <40% valley) satisfying EPA-1614 criteria for PBDE chromatographic separation. For the calculation of instrument limit of detection (IDL) and limits of quantitation (LOQ), the lowest concentration standard was serially diluted with n-nonane to between 0.5 -0.01 pg/μL and injected n=15 times. The IDL values were calculated using a two tailed student t-test at the 99% confidence interval and ranged from 2 to 100 fg on column for all 27-native mono-deca PBDEs (corresponding to 0.003–0.125 ng/kg in sample). Several food samples were tested for the PBDE content using triple quadrupole technology and versus GC-HRMS magnetic sector data show very close agreement. The low LOQs that are achievable using GC-MS/MS triple quadrupole technology are demonstrated in the case of BDE-49 in tallow and BDE-183 in reindeer; in both cases low ppt (ng/kg in extracted fat) results were reported with ion ratio % deviation from the calibration of <20%.

P-88 Simultaneous screening and quantitation solution for pesticides residues in milk by using unique GC-Orbitrap (Exactive) in full scan mode

Sarvendra P. Singh, Subodh Kumar Budakoti, Dasharath Oulkar, and Ashutosh Pathak

Thermo Fisher Scientific, Food Safety Solution Center, Ghaziabad, India; dasharath.oulkar@thermofisher.com

An accurate and robust method was optimized for the screening and quantitation of pesticides in milk by using gas chromatography based high-resolution mass spectrometry (GC-Orbitrap) in full scan mode. The use of high-resolution full-scan mass spectrometry is an emerging and popular analytical tool for accurate identification and confirmation of pesticide residues in food commodities. It is becoming more popular in food laboratories for determination. Day by day list of analytes keeps on increasing with respect to the consumer and regulatory's demand. Existing GC based triple quadrupole is performing till now with the targeted quantitative approach with limitations of the scan speed and resolving power to cover target as well as non-target simultaneously. To fulfill this requirement, a GC-Orbitrap could be one solution which addresses all the challenges. The data is being acquired in full scan mode which is taking care of increasing the number of active compounds and their metabolites within a single chromatographic run. Before the instrument analysis, sample preparation is equally important which can cover as many as possible analytes with their different physical and chemical properties. In this work, the European quick, easy, cheap, effective, rugged, and safe (EN QuEChERS) method was used. The potential of QuEChERS combined with GC-Orbitrap has been evaluated in terms of sensitivity and selectivity for milk. The target screening detection limit (SDL) was in the range of 1-2.5 ng/g for all the target analytes. Target analytes 148 (1.0 ng/g), 161 (2.5 ng/g) and 163 (5.0 ng/g) those fulfils the identification and confirmation criteria (< 5 ppm mass accuracy for precursor and/or product ion(s) with same RT in the extracted ion chromatograms) as per SANTE guideline. The method was quantitatively validated at 0.005, 0.01 mg/kg (default reporting limit) with 72-117% recoveries with <15 % RSD (precision). Overall, the optimized method is offering excellent sensitivity and selectivity for 163 analytes in milk by fulfilling the SANTE guideline in terms of validation as well as MRLs compliance with the European Union (EU) and Food Safety Standards Authority of India (FSSAI). This method provides a unique and quick screening and quantitation tool for the commercial food (Milk as test matrix) testing labs for testing pesticides and their metabolites without losing data quality and the possibility of retrospective analysis.

P-89 Extraction and Cleanup of Acrylamide in Complex Matrices Using Accelerated Solvent Extraction Followed by Liquid Chromatography Tandem Mass Spectrometry (LC-MS/MS)

Jeff Rohrer, Aaron Kettle, and Chris Shevlin

Thermo Fisher Scientific, Sunnyvale, CA

Acrylamide is formed during the cooking process of certain plant-based foods which are rich in carbohydrates and low in protein. Specifically, it forms when asparagine reacts with sugars such as glucose at high temperatures. Acrylamide was detected in fried foods by the Swedish National Food Authority in 2002. Since then, many food laboratories have successfully performed determinations for this compound on a variety of different food matrices. Acrylamide is a known carcinogen in animals.

Accelerated solvent extraction is an excellent technique for extraction of acrylamide from various fried food products; until recently, however, extraction of this compound from matrices such as coffee and chocolate has proven difficult. Traditional extraction techniques are time consuming and may cause bottlenecks in sample preparation. This study compares accelerated solvent extraction methods versus manual extraction procedures. This method combines the extraction of low levels of acrylamide from coffee and chocolate with an in-cell, solid-phase cleanup step. Acrylamide is then quantitated using LC/MS/MS.

P-90 Fast analysis of multi-class Pesticides panel in wine extracts using a Single Run LC-Triple Quadrupole Mass Spectrometry

Illaria Palini¹, Silvia Bani¹, Debora D'Addona², Charles T. Yang³, and Dipankar Ghosh³

¹ISVEA, Poggibonsi, Italy

²Thermo Fisher Scientific, Milan, IT,

³ **Thermo Fisher Scientific**, 355 River Oaks Parkway, San Jose, CA 95134, USA; charles.yang@thermofisher.com

Increasing food safety concerns and the growing agricultural trade has resulted in stringent pesticide regulations globally. To comply with such regulatory standards, screening methods for large numbers of pesticides is becoming important. Using liquid chromatography coupled Tandem quadrupole mass spectrometry offers highly sensitive, specific and selective detection in complex matrices for analysis of multi-class pesticides in food samples (wine and olive oil).

An Accucore aQ column was utilized for the separation of all analytes within 15minutes. Wine and olive oil were extracted with organic solvent using a simplified QuEChERS method and lipid removal cartridge to help remove excess fat or oil from olive oil and 1uL of sample was injected with a Vanquish Flex HPLC coupled to a TSQ Quantis triple quadrupole mass spectrometer. A multi-residue method was developed for screening (550+) and quantitation of approximately 300 pesticides in one 15-minute run with polarity switching. Ion ratios were used to confirm each analyte ($\pm 30\%$), plus accuracy of retention time to ± 0.1 min to show robustness of the method which are required for the EU SANTE Guidance 11813_2017. All pesticides analyzed show excellent Limits of Quantitation and Detection between 0.5 to 10ppb, while reproducibility (injection = 8/level) showed excellent precision and linearity with $R^2=0.9900$. Utilization of the lipid removal cartridge showed good %Rec between 10ppb and 50ppb between 70-120% which is within the SANTE Guidance. Unknown samples of wine and olive oil were also screened. Furthermore, the method was developed using software with built-in workflows for streamlining method development and routine analysis.

P-91 Maximization of Analytical Cannabis Extractions and Sample Clean-up through the use of a Single Process Combined Pressurized Fluid and Dispersive Solid Extraction (EDGE)

Patricia Atkins¹, Alicia Stell² & Brittany Leffler²

¹**SPEX CertiPrep**, 203 Norcross Rd; Metuchen, N.J. 08840; patkins@spex.com

²CEM Corporation, 3100 Smith Farm Rd; Matthews, NC 28104

The QuEChERS method has been shown to be practical for pesticide analysis on a number of different sample types and is increasingly being employed on more difficult matrices. Unfortunately some matrices either by

their nature or their economic value like cannabis can be difficult to analyze with just the QuEChERS method alone. These cannabis samples can show lower recoveries of pesticides and other target analytes than are often observed with more traditional agricultural products. An improved combined extraction and clean-up method is proposed in which both the extraction and dispersive solid phase extraction (DSE) steps are combined and heated using a pressurized fluid extraction and Adding heat and pressure to the process increases the efficiency leading to better sample clean-up and DSE improved analyte recoveries. In this study, a new combined extraction system was optimized to increase sample processing throughput, efficiency and recovery in a one-step process. Different analytes including pesticides, cannabinoids and terpenes were examined to determine improvement of recovery and method efficiency of the combined extraction apparatus. The new method showed marked improvement in sample clean-up, throughput and sample extraction recovery for cannabis testing.

P-92 Heavy Metal Exposure in Common Childhood Food Staples – The Peanut Butter & ‘Jelly’ Studies

Patricia Atkins,¹ Tina Restivo,² Robert Lockerman,² and Caryle McConnell¹

¹SPEX CertiPrep, 203 Norcross Ave, Metuchen, NJ 08840; patkins@spex.com

²CEM Corp. 3100 Smith Farm Rd, Matthews, NC 28104, US

Children are often known for their lack of food choices. But, many parents and caregivers know that there are usually some favorite childhood foods which are continuously part of a child’s diet, such as peanut butter and jelly sandwiches and raisins. Over the last few years, studies have found high levels of contamination in grapes and grape products such as juice and wine. Recent studies have been conducted showing the presence of arsenic in apple juices and wine. Arsenic based pesticides, particularly lead arsenate, were in widespread and use in the United States up until the final ban in 1988. Despite arsenic residue being recognized as a potential problem from the turn of the century, lead arsenate was one of the most widely used pesticides in the nation and was applied to millions of acres of crops through the 1940’s. Lead arsenate was the most commonly applied pesticide in fruit orchards, many still in use, so potential for arsenic contamination remains. Heavy metal pesticides were designed to be persistent and can cause environmental and health problems decades after being banned. In this study, samples were obtained of popular organic and regular raisins and peanut butter found in local supermarkets and stores. Samples were digested using microwave digestion and testing by ICP-MS to determine heavy metal contamination possible in these common childhood foods.

P-93 Analysis of Bisphenol A in Foods using Solid Phase Microextraction with an Overcoated Fiber

Stacy Shollenberger, Hillel Brandes, Justyce Jedlicka, Katherine K. Stenerson

MilliporeSigma, 595 Harrison Road; Bellefonte, PA 16803, USA; stacy.shollenberger@milliporesigma.com

Bisphenol A (BPA) is commonly used for food packaging applications such as polycarbonate bottles, and the linings of metal cans. It is a suspected endocrine disruptor, and thus low level long term exposure as a result of migration into food from packaging materials is a concern. Extraction methods for determination of BPA in food include both solvent and solid phase extraction, with the later more commonly used with liquid samples and the former for solid samples. Analysis can be done by either LC or GC, and both have been used throughout the literature. Solid phase microextraction (SPME) has been used for the determination of BPA in water but has not been widely used for food matrices due to sensitivity and fiber ruggedness issues associated with exposure to matrix components such as fats and proteins. In this work, the use of SPME was revisited in order to develop a quick, easy and sensitive method for analysis of BPA in a variety of food products. Matrix and fiber durability issues with immersion SPME were addressed through an overcoated (OC) divinylbenzene (DVB) fiber. The overcoating, which consists of polydimethylsiloxane (PDMS), protected the DVB layer from contamination, and increased the physical robustness of the fiber. SPME extraction using the OC-DVB fiber was followed by GC/MS/MS analysis for optimum sensitivity. The steps taken in method development and optimization will be described, as well accuracy in a variety of matrices. Data will also be presented on method ruggedness compared to a standard, non-overcoated DVB fiber.

P-94 Analysis of Polynuclear Aromatic Hydrocarbons in Paprika Powder Using EZ-POP NP SPE and a new Capillary GC Column

Hillel Brandes, Stacy Shollenberger, Justyce Jedlicka, Katherine K. Stenerson

MilliporeSigma, 595 Harrison Road; Bellefonte, PA 16803, USA; Hillel.brandes@milliporesigma.com

Paprika is a spice made from the drying of sweet peppers, and it is used for flavor and color in many types of cuisine. Contamination with polynuclear aromatic hydrocarbons (PAHs) can occur from environmental exposures to the pepper plants and/or during the drying process. The use of herbs in cooking and food production has become increasingly popular, thus exposure to PAHs, specifically those with carcinogenic properties, is of concern.

The sample preparation methods used in the testing of PAHs in herbs and spices require solvent extraction, followed by a cleanup step. The traditional cleanup methods that have been used include gel permeation chromatography (GPC), and solid phase extraction (SPE) with silica gel. A new approach for cleanup of high background samples to be analyzed for PAHs is a dual layer SPE cartridge containing Florisil[®] and Z-Sep/C18. This cartridge, available commercially under the name "EZ-POP NP", has been used for direct extraction and cleanup of edible oil samples analyzed for PAHs. In this work, PAHs were analyzed from paprika using an optimized QuEChERS extraction followed by cleanup with the EZ-POP NP SPE cartridge. Compared to QuEChERS cleanup, the EZ-POP NP yielded a much cleaner extract. Optimization of the extraction and EZ-POP NP cleanup procedures resulted in absolute recoveries of >70% at a spiking level of 10 ng/g. GC/MS/MS conditions were optimized to provide good peak shape and response, especially for the heavier 6 ring PAHs.

P-95 Analysis of Pesticides in Paprika - Development of an SPE Cleanup Method

Justyce Jedlicka, Stacy Shollenberger, Hillel Brandes, Katherine K. Stenerson

MilliporeSigma, 595 Harrison Road; Bellefonte, PA 16803, USA; Justyce.jedlicka@milliporesigma.com

Paprika is a spice made from the drying of sweet peppers, and is used for flavor and color in many types of cuisine. Pesticides applied to the peppers during cultivation can carry through the drying process, ending up in the dried paprika spice. Paprika is a commonly used spice, thus, in the interest of food safety, testing for the presence of pesticide residues is of great interest. For pesticide analysis, the "Quick, Easy, Cheap, Effective, Rugged and Safe" (QuEChERS) approach has become a popular method for extraction of various commodities, including spices. However, the background resulting from dried commodities can be problematic. Conventional QuEChERS cleanup may not be thorough enough for these types of samples. In this work, an SPE cleanup using a new dual layer, multi-sorbent cartridge was developed for cleanup of paprika extracts in the analysis of pesticide residues by LC/MS/MS and GC/MS/MS. The cartridge differs from conventional dual layer products containing carbon and PSA or aminopropyl silica in that it is much smaller, requiring less solvent for processing. It also contains blends of sorbents optimized to reduce oil and pigment background, while producing better pesticide recoveries than larger cartridges containing graphitized carbon black. The steps undertaken to develop the cleanup method for paprika extracts are described, and method accuracy and reproducibility are reported using replicates spiked at 50 ng/g with a variety of pesticides.

P-96 Use of Internal Degradation Marker Compounds for Large Multi-component Calibration Solutions

Daniel Biggerstaff, Ph.D., Huichen Stavros, Ph.D., Elliott Williams

O2Si Smart Solutions, an LGC Standards company, 7290B Investment Drive, North Charleston, SC 29418, USA; dan.biggerstaff@LGCGroup.com

Over the past few years, there has been a trend in the food safety industry towards developing multi-residue screening methods containing 500+ analytes in a single method. The greater the number of analytes in the calibration solution, the higher the probability of having analyte degradation in the calibration solution which can influence the quality of the data generated. It becomes important to be able to validate the quality of the working calibration solution used for daily calibration. We are developing a method of using internal marker

compounds in the working calibration solution to help validate that the concentration of analytes in the working solution has not degraded. This study shows the progress on how to implement an additional quality control step to increase confidence in data generated from these large multi-component solutions.

P-97 Seeing the whole picture: A multi-platform GC/MS screening approach for pesticides and environmental contaminants in food matrices

Anastasia Andrianova,¹ Melissa Churley,² Sofia Nieto,² Bruce Quimby,¹ Jessica Westland,¹ Courtney Milner²

¹Agilent Technologies, 2850 Centerville Rd, Wilmington, DE 19808; anastasia-andrianova@agilent.com

²Agilent Technologies, 5301 Stevens Creek Blvd, Santa Clara, CA 95051;

The increasing concern around trace-level food and environmental pollutants is driving the demand for more rapid and reliable methods for the identification of chemical residues. Meeting this challenge not only requires the fast deployment of technologies that can differentiate pesticides, PAHs, and other targets from organic interferences, but also requires the complex task of the analyst to search for hundreds of compounds in a wide variety of crop and environmental matrices. This presentation will discuss the application of three different GC/MS platforms for pesticide analysis in a selection of sixteen different brands of strawberries – the number one fruit in the Environment Work Group “Dirty Dozen” list for the last three years.

GC/MSD analysis utilized streamlined data processing with the Agilent’s MassHunter SureTarget automated deconvolution and library searching software. GC/MS/MS analysis employed targeting screening for trace level detection with the use of Agilent’s Pesticide and Environmental Pollutant MRM Database for over 1100 components. New streamlined screening workflow for GC/Q-TOF is based on a curated accurate mass database of pesticides and environmental contaminants and is designed to comply with SANTE and FDA guidelines. It offers high degree of flexibility of parameters settings for method optimization as well as the efficient data review process.

The benefits of each approach will be discussed in achieving high quality screening with confident quantitative capability.

P-98 Improvements in Quality and Throughput via Software Automation of Data Processing and Review

James B. Edwards and Randall K. Julian

Indigo BioAutomation; 385 City Center Drive, Suite 200; Carmel, IN 46032, USA; jedwards@indigobio.com

Despite dramatic improvements in the physical elements of chromatographic and/or mass spectrometric analysis, such as automated sample preparation methodologies or highly robust/performant instrumentation, the rate limiting step for the release of high confidence, high quality results is largely a factor of the methods of data review. This can be due to the highly complex sample matrices involved, along with the requirements to monitor for ever-decreasing levels of environmental contaminants, when acquired on a system under a relatively continuous, high throughput load. Advancements in data processing as well as automated quality assessment can make a significant impact on these practical, real-world factors in the laboratory.

A ‘before and after’ comparison is provided, from actual laboratory data (e.g. pesticide residue analysis in a cannabis matrix), which illustrates improvements in peak detection, compound identification, and quantitative accuracy/precision based on the use of robust statistical evaluation of the underlying raw data. The numbers and types of manual interventions (e.g. manual peak integration), and their resulting impacts on the results, are described in both discrete terms as well as trended data analytic views. Overall throughput time is also discussed.

Building on these data, potential quality improvement projects are identified, as well as an automated manner in which to assess any such changes in terms of method verification and validation. Method modifications which lower the technical and regulatory burden of data review are highlighted. In particular, method modifications (broadly considered: sample preparation, data acquisition, and/or processing techniques) which lower the frequency and magnitude of manual data intervention are described as a way to generate and release high quality data at a high operational tempo, while maintaining compliance with laboratory SOP and appropriate regulatory requirements.

P-99 Polycyclic Aromatic Hydrocarbons (PAH) analysis in fatty and complex food matrix using Gas Chromatography Triple Quadrupole Mass Spectrometry (GC/MS/MS)

Diana Wong, Limian Zhao, Bruce Quimby, Joerg Riener

Agilent Technologies, Inc. 2850 Centerville Road, Wilmington, DE, 19808, USA; diana.wong@agilent.com

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 sample preparation procedure using Enhanced Matrix Removal – Lipid (EMR) cleanup to remove matrix and lipid interferences for the investigation of low level PAHs in fatty and complex matrix. The challenge of analyzing PAHs by GC/MS/MS is due to the analyte's resistance to chemical reactions. PAHs accumulate rather than degrade and tend to de-sublime making PAHs difficult to vaporize. European Union-regulated PAHs were evaluated using a modified Gas Chromatograph Triple Quadrupole Mass spectrometry (GC/MS/MS) in Electron Ionization mode. The source to be kept clean from PAH and sample deposition by introducing a low flow of hydrogen during analysis. Post-run mid-column backflush provides a longer column lifetime by eliminating the need to bake out the column and less mass spectrometry maintenance by removing high-boiling matrix contaminants.

P-100 Nitrosamines analysis in drinking water using GC/MS/MS for Performance Equivalent to EPA Method 521

Diana Wong, Ron Honnold, Craig Marvin

Agilent Technologies, Inc. 2850 Centerville Road, Wilmington, DE, 19808, USA; diana.wong@agilent.com

Nitrosamines, particularly NDMA, are a group of disinfection byproducts frequently detected in finished drinking water and of concern to environmental agencies. The U.S. EPA Office of Groundwater and Drinking Water (OGWDW) developed Method 521 in 2004 to provide a procedure for trace level analysis of seven nitrosamines in finished drinking water by solid-phase extraction and chemical ionization tandem mass spectrometry (MS/MS). Ion Trap GC/MS is the approved technology, but the system is being obsoleted. Through an interlaboratory study, we show that migration to GC/MS/MS systems provided significant improvements in speed and sensitivity. This work demonstrates a GC/MS/MS method to allow for monitoring at levels below the current LCMRL (lowest concentration minimum reporting level) and detection limit set in Method 521. The GC/MS/MS method included the optimization of an additional nitrosamine, N-Nitrosomorpholine (NMOR). Three different laboratories collaborated to produce the LCMRL and performance data required for an Alternate Test Procedure method update. Results from these laboratories are compared to evaluate method feasibility and reproducibility. The method was validated by the three laboratories and written up for submission to the EPA for review. In 2008, the EPA issued a letter of equivalency deeming EEA 521.1 for the analysis of nitrosamines in drinking water by GC/MS/MS as a method that provided equivalent performance to Method 521.

P-101 A Multi-Residue LC-MS/MS Method for the Trace Analysis of 300 Pesticides Using a Solid-Core Stationary Phase With Unique C18 Bonding Chemistry

Geoffrey Faden¹ and Alan P McKeown²

¹ **MACMOD Analytical Inc.**, 103 Commons Court, PO Box 587, Chadds Ford, PA 19317 USA

² Advanced Chromatography Technologies Ltd, 1 Berry St, Aberdeen, Scotland

Pesticides are used globally as a method to eradicate unwanted pests to enhance crop yields and allow the more frequent re-use of land for crop growth. There are several classes of pesticides which all target different organisms: herbicides (plants), insecticides (insects), acaricides (mites/ticks) and fungicides (fungi). The use of pesticides is tightly regulated due to their potential toxicity. Pesticides can be harmful to humans if present in concentrations exceeding safe exposure limits. These limits (known as maximum residue limits – MRLs) have been put in place by the World Health Organisation (WHO), the Food and Agriculture Organisation of the United

Nations (FAO) and the European Food Safety Authority to ensure the safety of consumers worldwide. It is then the responsibility of individual countries to perform testing of agricultural samples to confirm pesticide residue levels are below the recommended thresholds. This testing is often performed using (U)HPLC coupled to tandem mass spectrometry to allow trace level determination.

This work presents an LC-MS/MS approach for the analysis of a 300 pesticide panel containing multiple classes of pesticides commonly found in foodstuffs and animal feed. A solid-core stationary phase with a unique and wide pH range stable C18 bonding chemistry was used for this multi-residue low level determination method.

P-102 The Detection of Fipronil and Fipronil Sulfone in Eggs

Jamie York,¹ Landon A. Wiest,² Justin Steimling,² Ty Kahler,² and Kevin A. Schug;²

¹The University of Texas at Arlington, 701 S. Nedderman Drive; Arlington, TX 76019, USA; jamie.schenk@mavs.uta.edu;

²Restek Corporation, 110 Benner Circle; Bellefonte, PA 16823, USA; landon.wiest@restek.com

Fipronil is a pesticide in the phenylpyrazole class and is used for a wide array of products including some home flea and tick preventatives/treatments for household pets.¹ The use of fipronil near animals for human consumption or laying hens is not permitted in Europe,² as fipronil is fat soluble and could contaminate meat and chicken eggs. However, millions of eggs were destroyed last year due to illegal use of fipronil in Europe near laying hens, which resulted in the contamination of millions of eggs with the insecticide.² Fipronil and its metabolite of similar toxicity, fipronil sulfone, inhibit the action of GABA in the central nervous system.¹ Fipronil is more effective at blocking the GABA action in insects than in mammals, but fipronil sulfone is less selective.¹ Once ingested Fipronil can cause hypertension, paralysis, and death in insects ^{1,3} and can cause indigestion, sweating, nausea, dizziness, agitation, vomiting, and seizures in humans.³ Because of the illegal use of fipronil around laying hens, it is crucial to develop a rapid, reliable, and sensitive method for detection of fipronil and its metabolite in eggs. In this study we optimized methods for extraction of fipronil from eggs using QuEChERS. We also evaluated multiple HPLC stationary phases and developed an optimized method calibrated from 0.1 to 10 ppb.

P-103 Contamination of turmeric with lead-containing pigments

Peter L. Morton¹, Mary E. Des Rosiers², Brent A. Summers³, Afi Sachi-Kocher¹, Vincent J. Salters¹

¹National High Magnetic Field Laboratory, 1800 E Paul Dirac Drive, Tallahassee, FL 32310, USA; pmorton@fsu.edu; ²Florida State University, Tallahassee, FL 32306, USA; ³University of South Florida-St. Petersburg, St. Petersburg, FL 33701, USA

A recent study in Bangladesh showed that 78% of 309 children aged 20-40 months had blood-lead (Pb) levels exceeding the CDC reference level of 5 µg/dL (Gleason et al., 2014). Lead-contaminated turmeric was identified as the most probable cause of the widespread poisoning, but a plausible mechanism of contamination was never conclusively determined. We reprocessed and reanalyzed subsamples of the original Gleason et al., 2014 turmeric samples to (1) independently validate the original Pb concentrations, (2) produce an expanded suite of potentially toxic trace elements (e.g., chromium, Cr), and (3) compare the Pb isotopic composition of the contaminated turmeric with those of anthropogenic and natural sources of Pb to the Indian Ocean. Our independent Pb concentration measurements showed excellent agreement (slope=0.9885, r²=0.982) with those of the original study. A multi-element reanalysis of the turmeric using HR-ICP-MS revealed that ~70% of the Pb in the turmeric was from lead chromate while the remaining 30% was from an unknown source containing Ba, possibly as barite (BaSO₄) from expired lead batteries. Both adulterants appear to have been mixed prior to adding to the turmeric, and all turmeric samples were adulterated with the same mixture, but at different relative amounts. The comparison of Pb isotopic compositions by multicollector ICP-MS showed that the turmeric Pb is different from Pb in industrial aerosols from the Bay of Bengal. All lines of evidence point towards intentional adulteration of turmeric with Pb-containing pigments, possibly added to improve the appearance and therefore value of an otherwise inferior turmeric product.

P-104 If Your Weed has Pesticide Contamination at 4000 Times the MRL, Should You Smoke it?

Philip L. Wylie and Jessica Westland

Agilent Technologies, Inc. , 2850 Centerville Rd., Wilmington, DE 19808, USA; phil_wylie@agilent.com

A high resolution, accurate mass GC/Q-TOF instrument was used to screen confiscated cannabis samples for more than 1000 pesticides and environmental contaminants. Each sample of ground dried cannabis flower was extracted in acetonitrile and the extract was passed through an endcapped C-18 SPE cartridge. Because cannabis extracts are so dirty, they were diluted 125:1 with solvent. The GC method used was retention-time-locked to a commercially available Personal Compound Database and Library (PCDL) that contains accurate mass spectra and locked retention times for 1020 compounds. To make data review easier the original PCDL was used to create a subset PCDL containing about 250 pesticides that are most commonly found on food commodities in the US. Data files were reviewed using two different procedures for finding suspect compounds – Find by Fragments (FbF) and Unknowns Analysis (UA). Twenty-one samples of confiscated cannabis flower were analyzed and eleven were found to be contaminated with detectable pesticide residues. Thirteen different pesticides were tentatively identified using these procedures. Concentrations of some of these pesticides were determined by calibration with standards. Two cannabis samples had pesticide levels that were estimated to be about 10 times greater than the highest EPA tolerance set for food and about 4000 times greater than the Canadian MRLs for dried cannabis. The two approaches to finding pesticides, FbF and UA, gave very similar results when analyzing the confiscated cannabis samples but data review was much easier using UA.

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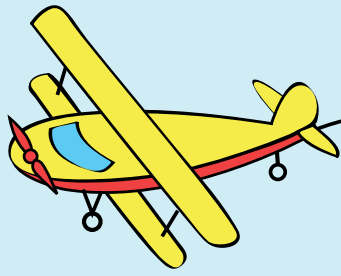
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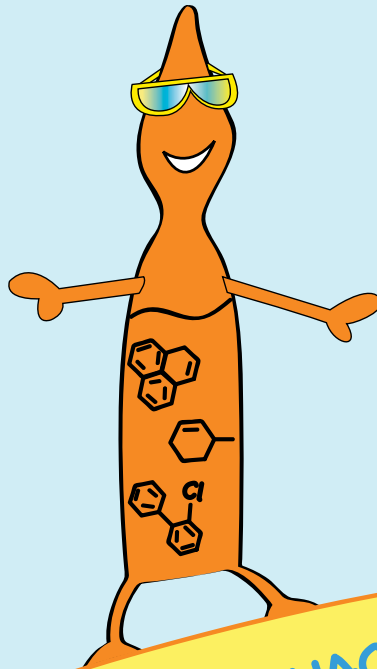
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