58TH ANNUAL

Online NACRW

NORTH AMERICAN CHEMICAL RESIDUE WORKSHOP

July 25-29, 2022

Table of Contents

FUTURE MEETING DATES

2023
July 23-26
Marriott Harbor Beach Resort
Fort Lauderdale, Florida

2024
July 14-17
Marriott Harbor Beach Resort
Fort Lauderdale, Florida

NACRW
NORTH AMERICAN
CHEMICAL RESIDUE WORKSHOP

Dear Attendees, Presenters, and Sponsors:

Welcome to the 58th North American Chemical Residue Workshop! We extend a warm greeting to our long-time attendees, our international guests, and our first time participants. We would like to especially thank our Sponsors for their generous support. Their financial contributions have made it possible for outstanding presentations, while maintaining affordable registration fees for attendees. The fantastic technical sessions, interactive poster presentations, and relaxed atmosphere have made NACRW a favorite event for many! While we can't meet in-person this year, we are looking forward to the many presentations and opportunities to interact virtually.

We hope you will make plans to attend the NACRW Reference Materials and Veterinary Drugs Working Groups on Monday, July 25. Each Working Group Session has a number of interesting presentations planned, as well as an open forum to discuss questions and answers.

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 which include: MS-based Screening, Quantitation and Identification Techniques for Chemical Contaminants Analysis in Food, Advances in Sample Prep, Method Development and Validation for Multi-Residue Methods, QA for Pesticides Analysis, Environmental Contaminants and Toxins, Troubleshooting / Challenges and Solutions, Advances and Issues in Analytical Methods for Veterinary Drug Residues, and Common Challenges with Cannabis Compliance Testing. In addition to these topics, there will be the Troubleshooting Forum which allows attendees to ask questions to our panel of experts. If you have a question, please submit your question at registration or through the chat feature in the virtual platform. New to this year's program is our Speed Networking. During the Speed Networking, people will be randomly grouped and will have the opportunity to meet fellow conference goers for a set time period. This is great opportunity to network, share stories, and meet Scientists in the community.

In addition to our oral sessions, please plan to attend the poster sessions and vendor technical presentations. The poster authors will be presenting their posters at designated times listed in the program. This is a great opportunity to engage the authors and ask questions. 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 will be available at their virtual posters during designated times to have conversations and answer questions. We are pleased to offer Technology Presentations between sessions during the week. This is a great opportunity to hear about the latest developments and exciting new products or applications from some the sponsors.

I would like to thank the fantastic volunteers who have helped make this virtual event a reality. To the 2022 NACRW Organizing Committee, Program Committee, especially Julie Kowalski, Jessica Krank, and Executive Director, Teri Besse; it has been a pleasure to work with you, 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.

We hope you enjoy your time at NACRW!

Sincerely,

Alexandria Bush, 2022 Organizing Committee President
Julie Kowalski and Jessica Krank, 2022 Program Committee Co-Chairs
2022 Organizing Committee and Program Committee Members

Board of Directors

Sherry Garris, Chair
Jack Cochran
Jo Marie Cook, Treasurer
Susan Eigen
Brian Eitzer
Simon Hird
Julie Kowalski
Gail Parker
Sherri Turnipseed
Jon Wong
Teri Besse, Executive Director

Program Committee

Co-Chairs:
Julie Kowalski, jkSS, LLC
Jessica Krank, USEPA-NEIC

Co-Chairs Elect:
Kenneth Kise,Iowa Dept, of Agriculture
Susie Genualdi, US FDA

Immediate Past Co-Chairs:
Katie Carlos, US FDA CFSAN
Mike Filigenzi, CAHFS, U.C. Davis

Organizing Committee Officers

President:
Alexandria Bush, Restek Corporation

Secretary:
Ryan Undeen, Mérieux Nutrisciences

President-Elect:
Oscar Cabries

Program Committee Members

Jens Andersen, Botswana Int'l University
Brian Eitzer, The Conn. Agr. Exp. Station
Amadeo Fernandez-Alba, University of Almeria
Stephen Goldman,Kaycha Labs
Wiley Hall, Safe Food Alliance / DFA of CA
Jana Hepner,Restek Corporation
Simon Hird,Waters Corporation
Kenneth Kise,Iowa Dept, of Agriculture
Scott Krepich,SCIEX
Alex Krynitsky,Symbiotic Research LLC
Katerina Mastovska,Eurofins
Sareeta Nerkar,Pickering Laboratories
Matt Noestheden,SCIEX
Raegyn Taylor,USDA
Sherri Turnipseed,US FDA-Animal Drugs Research Center
Eric Verdon,ANSES
Jian Wang,Canadian Food Inspection Agency
Wendy Young,Food and Drug Administration

Organizing Committee Members

Patricia Atkins, SPEX
Kevin Armbrust, Louisiana State University
Rodney Bennett
André De Kok
Brittany Holmes, Central Washington University
Nathan Johnson, Campbell Soup
Joe Konschnik, Restek Corporation
Martha Maier, Vista Analytical Laboratory
Yelena Sapozhnikova, USDA ARS
Jon Wong, USFDA
Lin Ye, Food and Drug Administration

Poster Committee

Co-Chairs:
André de Kok, formerly of Waigeningen Food Safety Research
Brittany Holmes, Central Washington University

Poster Committee Members

Brian Eitzer, The Conn. Agr. Exp. Station
Raegyn Taylor, USDA

to all of our amazing volunteers!

Thank you to our Conference Sponsors!

Platinum Sponsors

Agilent

RESTEK

SCIEX

ThermoFisher SCIENTIFIC

Waters

THE SCIENCE OF WHAT'S POSSIBLE.™

Thank you to our Conference Sponsors!

Friends of NACRW

BIPEA

CANNABIS
science and technology
Advancing Research, Quality, and Education

LCGC®

Spectroscopy®

Thank you to the NACRW Reference Materials Working Group Sponsors

ADPEN LABORATORIES INC.

Lab Instruments
high pure standards & chemicals division

RESTEK

SCIEX

spex® an antylia scientific company

ThermoFisher SCIENTIFIC

Vendor Technology Presentations

Monday, July 25, 2022
9:30-10:30 am
Restek Corporation Technology Presentation

Novel Approaches for Optimizing Workflows for Multiresidue Pesticides and Mycotoxins
Nathaly Reyes-Garcés, **Ph.D.**, Scientist III, LC Solutions-Applications, Restek, and **Jana Hepner, Ph.D.,** Senior Scientist I, GC Solutions-Applications, Restek, Bellefonte, PA; USA

Two workflow solutions will be presented for the analysis of pesticide residues and mycotoxins in hemp and foods. First, a complete workflow for analysis of both pesticides and mycotoxins in hemp and will include a variety of sample preparation approaches followed by a combination of both LC and GC-MS/MS analyses. Recovery studies using hemp and cannabis edibles will demonstrate the effectiveness of these workflows. Second, an evaluation of applying Concurrent Solvent Recondensation Large Sample Volume Splitless injection (CSR-LVSI) in combination with Low Pressure GC-MS (LPGC-MS) as a way to improve sensitivity by overcoming the limitation of a conventional 1-2uL injection for pesticide analysis.

Impacts on detection limits and other parameters resulting from using various solvents and injection volumes will be discussed.

Monday, July 25, 2022
12:45-1:45 pm
Thermo Fisher Scientific Technology Presentation

Implementing new GC-MS technology to stay ahead with your pesticides analysis
Adam Ladak, Product Marketing Manager, GC Quadrupole MS, Thermo Fisher Scientific; Hemel Hempstead; UK; Adam.Ladak@thermofisher.com

During this presentation, the capabilities of the new Thermo Scientific GC-MS/MS system, which was launched in March 2022, will be discussed. The system design was based on direct customer feedback in order to overcome challenges faced by analytical testing laboratories. The new system provides sensitivity to meet the strictest regulatory limits and has modular GC for increased flexibility in system configuration, while including unique maintenance options and compatibility with online automated clean-up to minimize instrument downtime and increase sample throughput. An overview of applications on the new GC-MS/MS with data examples will be given, including the analysis of pesticide in baby food and the determination of ethylene oxide in food, showing how your laboratory can stay ahead with unstoppable confidence. At the conclusion of the presentation, our application expert will answer any questions in a live Q and A.

Key Learning Objectives:
• Learn how the new feature of the GC-MS/MS systems can enable your laboratory to be more productive.
• Learn how regulatory analytical laboratories can utilize the new systems to overcome their challenges in food applications.
• Learn the benefits of implementing the new systems in your laboratory and how a rapid return on investment can be achieved.

Tuesday, July 26, 2022
12:30-1:30 pm
Agilent Technology Presentation

Enabling a Fast and Robust GC/MS/MS Pesticide Analysis with Helium and Hydrogen Carrier Gasses
Anastasia Andrianova, GC/MS Applications Scientist, Agilent Technologies; Wilmington, DE; USA; anastasia.andrianova@agilent.com

This presentation will describe achieving fast GC/MS/MS analysis, while maintaining robust system performance in complex food matrices. Two system configurations using mid-column backflush provide robust analysis in 10 minutes while maintaining sufficient chromatographic resolution for the analysis of 203 compounds. A conventional 15m x 15m (0.25mm x 0.25µm) column configuration was used with helium carrier gas and a narrow bore 10m x 10m (0.18mm x 0.18µm) was used with helium and hydrogen carrier gasses. A novel EI source was used for pesticides analysis with hydrogen carrier gas. It is optimized to address undesired in-source hydrogen-related issues like hydrogenation and provides improved performance with hydrogen carrier gas in chemical residue analysis with GC/MS.

Key Learning Objectives:
- Optimized GC/MS column configurations enabling fast and robust pesticides analysis compatible with helium and hydrogen carrier gasses
- Novel EI source designed to address undesired in-source hydrogen-related issues
- Best practices to unlock maximum performance in analysis of over 200 pesticides in challenging food matrices by GC/MS/MS

Wednesday, July 27, 2022
12:30-1:30 pm
SCIEX Technology Presentation

Improving Laboratory Efficiency with Sensitivity - A Residue Mega Method Case Study
Craig Butt, Ph.D., Manager, Applied Markets, SCIEX; craig.butt@sciex.com

In an ideal scenario, a single sample preparation and analysis method would be employed for all contaminants in all food and feed matrices that must be analyzed by a particular laboratory. This is often not possible due to matrix effects and/or sensitivity requirements, resulting in a reduction in laboratory efficiency and inflated operating costs. Here, a single sample preparation was used to analyze hundreds of pesticides, veterinary drugs and mycotoxins in different food and feed matrices in a single injection using the SCIEX 7500 system. High instrument sensitivity permitted the use of a dilution strategy with QuEChERSER extraction to mitigate matrix effects while maintaining the required quantification limits. This work explores the tradeoffs between dilution and sensitivity requirements, with the goal of simplifying sample preparation procedures and streamlining laboratory workflows.

Thursday, July 28, 2022
12 noon-1:00 pm
Waters Corporation Technology Presentations

New Product Innovations to Advance Food Safety Testing
Emily Rose Britton, Sr. Market Development Manager, Americas Food & Natural Products, Waters Corporation; Emily_Britton@waters.com

Growing population and increasing global demand for food have increased the regulatory requirements and workload for food testing labs. New analytical tools and technologies are needed to keep up with demands for better productivity, compliance and throughput. Join us to learn about the latest developments in chromatography, mass spectrometry, sample prep automation, software and services at Waters to gain efficiency across your food testing workflows.

Routine Quantitation of Challenging Compounds using Xevo™ TQ Absolute
Gordon Fujimoto, Group Leader, Chemical Analysis Demo Lab, Waters Corporation; Gordon_Fujimoto@waters.com

Routine analysis of challenging negative ionizing compounds such as anionic polar pesticides and PFAS has become a requirement for many food testing laboratories. The demand for lower limits of quantification (sub µg/kg) for these contaminants can be addressed with the enhanced negative ion sensitivity of the Xevo™ TQ Absolute system. An incredibly compact, robust and sustainable tandem mass spectrometer that can enable dilute and shoot workflow and reduce injection volume to reduce matrix load on the LC-MS/MS system can help your lab achieve time and cost savings while delivering reproducible results.

MEETING PROGRAM

Monday, July 25, 2022

9:00-9:05 am	**Welcome Remarks: Sherry Garris**, Chair, FLAG Works, Inc./NACRW
9:10-9:20 am	**Speed Networking**
9:30-10:30 am	**Restek Corporation Technology Presentation** **Novel Approaches for Optimizing Workflows for Multiresidue Pesticides and Mycotoxins** **Nathaly Reyes-Garcés**, Ph.D., Scientist III, LC Solutions-Applications, Restek, and **Jana Hepner**, Ph.D., Senior Scientist I, GC Solutions-Applications, Restek, Bellefonte, PA; USA
10:45 am-12:15 pm	**NACRW Veterinary Drugs Working Group**
10:45-11:05 am **VD-01**	**Maïwenn Le Floch**, ANSES, Javené; France **Progress of the VDR Collaborative Study**
11:05-11:25 am **VD-02**	**Steven Lehotay**, USDA Agricultural Research Service, Wyndmoor, PA; USA **Stability of Veterinary Drugs**
11:25-11:45 am **VD-03**	**Eric Verdon**, ANSES, Fougeres, France **Insights into the new EU Implementing Regulation for Analytical Methods Performance for Veterinary Drug Residue Official Control in Food: CIR (EU) 2021/808**
11:45 am-12:15 pm **VD-04**	**Sherri Turnipseed**, USFDA-Animal Drugs Research Center, Denver, CO; USA **Open Forum: Question and Answer Session for NACRW Veterinary Drugs Working Group**
12:45-1:45 pm	**Thermo Fisher Scientific Technology Presentation** **Implementing new GC-MS technology to stay ahead with your pesticides analysis** **Adam Ladak**, Product Marketing Manager, GC Quadrupole MS, Thermo Fisher Scientific; Hemel Hempstead; UK; Adam.Ladak@thermofisher.com
2:00-4:00 pm	**NACRW Reference Materials Working Group**
2:00-2:20 pm **RM-01**	**Patricia Atkins**, Spex, An Antylia Scientific Company, Metuchen, NJ; USA **Increasing the Scope of the Reference Materials Use in Trace Analysis: Highlights forthe Second Edition**
2:20-2:40 pm **RM-02**	**Kevin Kubachka**, US FDA - Forensic Chemistry Center, Cincinnati, OH; USA **Addition of Inorganic Guidance to the Reference Material Use in Trace Analysis Manual**
2:40-3:00 pm **RM-03**	**Sidney Sudberg**, Alkemist Labs, Garden Grove, CA; USA **Use of Reference Materials, for the Authentication of Natural Products, Botanicals, and Other Agricultural Materials**
3:00-3:20 pm **RM-04**	**Joe Konschnik**, Restek Corporation, Bellefonte, PA; USA **Botanicals & Dietary Supplements Collaboration Opportunities**
3:20-4:00 pm **RM-05**	**Jo Marie Cook**, Florida Department of Agriculture and Consumer Services, Venice, FL; USA; and **Patricia Atkins**, Spex, An Antylia Scientific Company, Metuchen, NJ; USA **Open Forum: Question and Answer Session for NACRW Reference Materials Working Group**

Tuesday, July 26, 2022

9:00-9:10 am — **Opening Remarks**
Alexandria Bush, President, 2022 NACRW Organizing Committee

9:10-10:10 am — **Poster Session Presentations**

10:15 am-12:05 pm — **SESSION 1:** sponsored by ThermoFisher SCIENTIFIC
MS-based screening, quantitation and identification techniques for chemical contaminants analysis in food
Co-Chairs: Jian Wang and Amadeo R. Fernández-Alba

10:15 -10:20 pm — **Session Sponsor Recognition – Thermo Fisher**

10:20-10:40 am — **Jon Wong**, US FDA, College Park, MD; USA
O-01 — **Evaluation of a non-Target Data Acquisition for Target Analysis (nDATA) Workflow for Analysis of 1200 Pesticides in Fresh Produce using Liquid Chromatography-High Resolution Accurate Mass Spectrometry with a Compound Database**

10:40-11:00 am — **Yufang Zheng**, National Institute Of Standards And Technology, Gaithersburg, MD; USA
O-02 — **Enhancing the Coverage and Quality of Spectra of Pesticides in a Comprehensive Electron Ionization (EI) Mass Spectral Library**

11:00-11:20 am — **Amadeo Fernández-Alba**, EURL-FV University of Almería, La Cañada de San Urbano, Almería; Spain
O-03 — **Analysis of high polar pesticides in fruits and vegetables by various chromatographic approaches- A critical evaluation**

11:20-11:40 pm — **Alexander Semyonov**, Thermo Fisher Scientific, Austin, TX; USA
O-04 — **Cationic polar pesticides in food and beverage commodities by IC-MS/MS**

11:40– 12:05 pm — **Live Q&A for Session 1**

12:30-1:30 pm — **Agilent Technology Presentation**
Enabling a Fast and Robust GC/MS/MS Pesticide Analysis with Helium and Hydrogen Carrier Gasses
Anastasia Andrianova, GC/MS Applications Scientist, Agilent Technologies; Wilmington, DE; USA; anastasia.andrianova@agilent.com

2:00-4:05 pm — **SESSION 2:** sponsored by RESTEK
Advances in sample prep
Co-Chairs: Sareeta Nerkar and Jana Hepner

2:00-2:05 pm — **Session Sponsor Recognition – Restek Corporation**

2:05-2:30 pm — **Anton Kaufmann**, Official Food Control Authority of The Canton Of Zurich, Zürich; Switzerland
O-05 — **Improving the quechers liquid/liquid extraction of analytes with widely varying physicochemical properties: Example of 201 veterinary drugs in milk**

2:30-2:55 pm — **Nicolas Michlig**, USDA, Wyndmoor, PA; USA
O-06 — **Assessment of a new cartridge design in automated miniaturized solid-phase extraction cleanup for pesticides and environmental contaminants in fatty and nonfatty matrices**

2:55-3:20 pm **Jian Wang**, Canadian Food Inspection Agency, Calgary, Alberta; Canada
O-07 **The Study of Ultra-high Performance Liquid Chromatography Electrospray Ionization Q-Orbitrap Mass Spectrometry and Various Extraction Methods for Fingerprinting and Identification of Molecular Authenticity Markers from Apple Juices and Grape Juices**

3:20-3:45 pm **Jinchuan Yang**, Waters Corporation, Milford, MA; USA
O-08 **Analysis of Aminoglycosides in Foods using LC-MS/MS with a Zwitterionic Stationary Phase Column**

3:45-4:05 pm **Live Q&A for Session 2**

4:10-4:30 pm **Speed Networking**

Wednesday, July 27, 2022

9:00-10:00 am **Poster Session Presentations**

10:05-12:05 pm **SESSION 3:** **sponsored by** Waters THE SCIENCE OF WHAT'S POSSIBLE.™
Method development and validation for multi-residue methods
Co-Chairs: Kate Mastovska and Raegyn Taylor

10:05-10:10 am **Session Sponsor Recognition – Waters**

10:10-10:25 am **Simon Hird**, Waters Corporation, Wimlsow, Cheshire; United Kingdom
O-09 **Bringing reliable determination of anionic polar pesticides in food to the routine laboratory**

10:25-10:40 am **Virgínia Cruz Fernandes,** REQUIMTE/LAQV, Instituto Superior de Engenharia, Instituto Politécnico do Porto, PORTO; Portugal
O-10 **The use of honey as an environmental biomonitor of contamination in Natural Park of Montesinho: a comparison of extraction methods for the determination of multiple contaminants**

10:40-10:55 am **Anastasia Andrianova**, Agilent Technologies, Wilmington, DE; USA
O-11 **A Robust High-Throughput GC/MS/MS Analysis of 203 Pesticides in Under 10 Minutes with Helium and Hydrogen Carrier Gasses**

10:55-11:10 am **Shun-Hsin Liang,** Restek Corporation, Bellefonte, PA; USA
O-12 **Simultaneous Determination of Alternaria Toxins, Ergot Alkaloid Epimers, and Other Major Mycotoxins in Various Food Matrices by LC-MS/MS**

11:10-11:25 am **Carlos Parra**, Now Foods, Bloomingdale, IL; USA
O-13 **Robust Analysis of Pesticide Residues in Black Tea by Improved GC-MS/MS**

11:25-11:40 pm **Michael Gross**, US Geological Survey, Sacramento, CA; USA
O-14 **Development and Application of a Multiresidue Method for the Analysis of Pesticides in Plasma**

11:40-12:05 pm **Live Q&A for Session 3**

12:30-1:30 pm — **SCIEX Vendor Technology Presentation**
Improving Laboratory Efficiency with Sensitivity - A Residue Mega Method Case Study
Craig Butt, Ph.D., Manager, Applied Markets, SCIEX; craig.butt@sciex.com

1:45-1:55 pm — **Speed Networking**

2:00-4:00 pm — **SESSION 4:**
QA for pesticide analysis
Co-Chairs: Jens Andersen and Brian Eitzer

2:00-2:25 pm
O-15 — **Daniel Biggerstaff**, o2si Smart Solutions, North Charleston, SC; USA
What ISO 17034 Accreditation of Reference Materials Mean for Your Quality Assurance/ Quality Control Program

2:25-2:50 pm
O-16 — **Jens Enevold Thaulov Andersen**, Botswana International University of Science and Technology, Palapye; Botswana
The Principle of Pooled Calibrations Indicates Shrinking the Valid Range of pH Measurements

2:50-3:15 pm
O-17 — **Jonathan Decenzi**, NOW Foods, Bloomingdale, IL; USA
Challenges in Determining Acceptability of Dietary Supplement Raw Materials Based on Pesticide Residue Results Obtained Using In-House Methodology

3:15-3:40 pm
O-18 — **Jens Enevold Thaulov Andersen**, Botswana International University of Science and Technology, Palapye; Botswana
Squeezing more out of Laboratory Analyses with QA/QC, Appetizer to short course in 2023

3:40-4:00 pm — **Live Q&A for Session 4**

Thursday, July 28, 2022

9:00-10:00 am — **Poster Session Presentations**

10:05-11:50 am — **SESSION 5**:
Environmental contaminants and toxins
Co-Chairs: Ken Kise and Wendy Young

sponsored by SCIEX

10:05-10:10 am — **Session Sponsor Recognition – SCIEX**

10:10-10:30 am
O-19 — **Kai Zhang**, US Food and Drug Administration, College Park, MD; USA
Determination of mycotoxins in dried fruits using LC-MS – homogeneity, troubleshooting, and confirmation of identity

10:30-10:50 am
O-20 — **Raegyn Taylor**, USDA, Wyndmoor, PA; USA
Validation of QuEChERSER for PFAS in Food with Method and Instrumental Comparisons

10:50-11:10 am
O-21 — **Amos Dwamena**, Smithers, Wareham, MA; USA
Pesticides Exposure Assessment: Analytical Method Challenges and Opportunities

11:10-11:30 am
O-22 — **Michelle Hladik**, U.S. Geological Survey, Sacramento, CA; USA
Measuring Wild Bee Exposure to Pesticides Using Silicone Bands and Tissue Residues

11:30-11:50 am — **Live Q&A for Session 5**

12 noon-1:00 pm — **Waters Corporation Technology Presentations**

New Product Innovations to Advance Food Safety Testing
Emily Rose Britton, Sr. Market Development Manager, Americas Food & Natural Products, Waters Corporation; Emily_Britton@waters.com

Routine Quantitation of Challenging Compounds using Xevo™ TQ Absolute
Gordon Fujimoto, Group Leader, Chemical Analysis Demo Lab, Waters Corporation; Gordon_Fujimoto@waters.com

1:05-2:35 pm — **SESSION 6:**
Troubleshooting/Challenges and Solutions
Co-Chairs: Alex Krynitsky and Wiley Hall

1:05-1:30 pm — **Charles Powley**, Center For PFAS Solutions, New Castle, DE; USA
O-23 — **PFAS Analytical Method Development and Troubleshooting**

1:30-1:55 pm — **Patricia Atkins**, Spex, An Antylia Scientific Company, Metuchen, NJ; USA
O-24 — **Chemistry, Chromatography & Cannabis: Troubleshooting the Cannabis Workflow for Analysis**

1:55-2:20 pm — **Sarah King**, Eurofins Central Analytical Laboratories, New Orleans, LA; USA
O-25 — **Pesticide Residue Testing: Challenges Faced with Routine Analysis**

2:20-2:35 pm — **Live Q&A for Session 6**

2:45-4:00 pm — **SESSION 7:**
Forum Troubleshooting
Moderators: Simon Hird and Scott Krepich

Panelists: Charles Powley, Center For PFAS Solutions, New Castle, DE; USA; **Patricia Atkins**, Spex, An Antylia Scientific Company, Metuchen, NJ; USA; **Sarah King**, Eurofins Central Analytical Laboratories, New Orleans, LA; USA; **Sherri Turnipseed**, USFDA, Denver, CO; USA; and **Brian Veach**, USFDA, Jefferson, AR; USA

4:05-4:30 pm — **Speed Networking**

Friday, July 29, 2022

9:00-10:45 am — **SESSION 8:**
Advances and Issues in Analytical Methods for Veterinary Drug Residues
Co-Chairs: **Eric Verdon and Sherri Turnipseed**

9:00-9:20 am — **Jessica Rafson**, FDA, Denver, CO; USA
O-26 — **Evaluation of coated blade extractions prior to LC-HRMS for the analysis of veterinary drug residues in aquaculture products**

9:20-9:40 am — **Randall Purves**, Canadian Food Inspection Agency, Saskatoon, Saskatchewan; Canada
O-27 — **Development of a confirmatory method for the detection of gestagens in animal liver using a simplified extraction with LC-MS and high-field asymmetric waveform ion mobility spectrometry (FAIMS)**

9:40-10:00 am	**Ghinwa Ismail**, Anses, French National (NRL) and EU Reference Laboratory (EU-RL) for Veterinary Medicinal Product and Antimicrobial Residues in Food from Animal Origin, Laboratory of Fougères; and CNRSL, Lebanese Atomic Energy Commission, Laboratory for Analysis of Organic Compounds, Lebanon, Fougères, Bretagne; France
O-28	**Optimization of multi-residues screening method for the detection of antibiotics in Milk products: the case of Labneh**
10:00-10:20 am	**Ryan Gibbs**, Canadian Food Inspection Agency, Dartmouth, Nova Scotia; Canada
O-29	**Evaluation of the uncertainty in differing approaches to correct for matrix effects in LC-MS/MS analysis of antibiotics and growth promoters in fish products**
10:20-10:45 am	**Live Q&A for Session 8**
11:15 am–12 noon	**Media Partner Presentation: A Closer Look at Cannabis Science: Unmet Needs, Trends & Thoughts** **Josh Crossney,** Founder, Cannabis Science Conference and Director, MJH Life Sciences
12:20-12:30 pm	**Speed Networking**
12:35-2:40 pm	**SESSION 9: sponsored by Agilent** **Common Challenges with Cannabis Compliance Testing** **Co-Chairs: Matt Noestheden and Stephen Goldman**
12:35-12:40 pm	**Session Sponsor Recognition – Agilent**
12:40-1:00 pm	**Lukas Vaclavik**, Eurofins Food Chemistry Testing, Madison, WI; USA
O-30	**Low Level LC-MS/MS Determination of Cannabinoids in Isolates, Hemp Plant Materials, and Extraction By-Products**
1:00-1:20 pm	**Anthony Macherone**, Agilent Technologies, Wilmington, DE; USA
O-31	**The Pros and Cons of Mass Spectrometry for Cannabis Potency Testing**
1:20-1:40 pm	**Walter Wilson**, NIST, Gaithersburg, MD; USA
O-32	**Results from two CannaQAP Interlaboratory Study Exercises at NIST for Improving Cannabinoid, Toxic Element, and Moisture Measurements in the Cannabis Industry**
1:40-2:00 pm	**Kate Evans**, Longboard Scientific Consulting Corporation, Colorado Springs, CO; USA
O-33	**Navigating Unchartered Territory: Cannabis Testing Landscape from an Honest Perspective**
2:00-2:20 pm	**Avinash Dalmia**, Perkinelmer, Shelton, CT; USA
O-34	**A novel LC/MS/MS method with ESI and APCI ion source for analysis of 102 pesticides and 5 mycotoxins regulated by Colorado state in Hemp**
2:20-2:40 pm	**Live Q&A for Session 9**
2:40-3:00 pm	**Poster Awards and Closing**

ORAL PRESENTATION ABSTRACTS

O-01

Evaluation of a non-Target Data Acquisition for Target Analysis (nDATA) Workflow for Analysis of 1200 Pesticides in Fresh Produce using Liquid Chromatography-High Resolution Accurate Mass Spectrometry with a Compound Database

Jon W. Wong[1], Jian Wang[2], Willis Chow[2], and Roland Carlson[3]

[1]US Food and Drug Administration, Center for Food Safety and Applied Nutrition (US FDA CFSAN), College Park MD USA
[2]Canadian Food Inspection Agency (CFIA), Calgary Laboratory, Calgary AL Canada
[3]California Department of Food and Agriculture, Center for Analytical Chemistry (CDFA CAC), Sacramento CA USA

Pesticide laboratories face enormous challenges such as the analysis of an increasing number of pesticides and difficult food commodities with limited resources and time. A compound database (CDB) based on the retention times and theoretical masses of fragments from high resolution MS/MS spectra of 1200 pesticides and metabolites was developed for identification. The CDB is evaluated and validated for analyzing pesticides in fruits and vegetables using QuEChERS and ultrahigh performance liquid chromatography-quadrupole Orbitrap mass spectrometry operating in full scan MS/variable data independent acquisition MS/MS mode by CFIA Calgary Laboratory, US FDA CFSAN, and CDFA CAC. Identification of pesticides followed Codex, SANTE and FDA guidelines by determining retention times (± 0.5 min) of the precursor ion (RTP, $\delta_M \leq \pm 5$ ppm) and fragment ions (RTFI, $\delta_M \leq \pm 10$ ppm). Evaluation of the nDATA workflow was based on the determination of the false positives and negatives of all identified pesticides in unfortified and fortified (10 and 100 µg/kg) produce matrices. Analysis of incurred residues from archived produce samples were also investigated among the laboratories. This approach can be used to screen 1200 pesticides and quantify the ~250 most frequently incurred residues in various produce commodities, further improving monitoring capabilities for pesticide analysis.

O-02

Enhancing the Coverage and Quality of Spectra of Pesticides in a Comprehensive Electron Ionization (EI) Mass Spectral Library

Yufang Zheng; Edward Erisman; Weihua Ji; Tytus Mak; Lewis Y. Geer; Stephen Stein; William Wallace

Mass Spectrometry Data Center, National Institute of Standards and Technology, Gaithersburg, MD 20899, USA; Yufang.zheng@nist.gov

A high-quality, comprehensive reference EI mass spectral library provides a fast and reliable way to assist the identification of pesticides. Here we report a novel procedure to expand a NIST EI mass spectral library for insecticides, fungicides herbicides and related compounds of environmental concern. The procedure involves targeting pesticides of interest for purchase with a computer program. A list of 3374 pesticides from the PAN pesticides database was first generated by a computer program to produce a list of 1662 unique compounds with valid CAS#s, followed by analysis of spectra for these compounds already present in the NIST2020 library to via a Python program. Results show that 44 % of those compounds are not present in the NIST20 EI library, 20 % are present but not measured by NIST, and 10 % are present but not of the highest quality. Comparison of these compounds to commercially available compound information served to make a final purchase list. So far, we developed the initial and silylated derivatization protocols to acquire high quality spectra out of 524 pesticides by GC/MS. A total of 250 pesticides with varying functional groups were derivatized. Data processing includes the extraction of spectra from raw data using AMDIS and the application of new evaluation algorithms (MS Interpreter, structure similarity search or hybrid search) to confirm the identity of pesticides. So far, 1070 high- quality pesticide spectra out of 524 pesticides have been acquired and added to the upcoming NIST2023 library.

O-03

Analysis of high polar pesticides in fruits and vegetables by various chromatographic approaches: A critical evaluation

Amadeo R. Fernández-Alba

University of Almería, Department of Physics and Chemistry, Agrifood Campus of International Excellence (ceiA3), La Cañada de San Urbano, 04120, Almería, Spain; amadeo@ual.es

Highly polar pesticides are frequently used in agriculture. However, their physicochemical properties make very difficult the analysis of these compounds following common procedures. Polar pesticides show poor retention and peak shapes in the common stationary phases used for multiresidue methods of pesticides. For this reason, ion chromatography or specifically design RPLC columns with different stationary phases have been developed to perform the analysis of these particular compounds.
In the present work various approaches based on ion (IC) and hydrophilic interaction liquid chromatography (HILIC) are evaluated looking for efficient solutions to routine analysis of these residues with different matrices.
Ion chromatography represent a very good alternative for all residues under study ((chlorate, perchlorate, fosetyl-aluminum, glyphosate, aminomethylphosphonic acid (AMPA), phosphonic acid, N-acetyl AMPA, and N-acetyl glyphosate) reaching LOQ around 0.01 mg/kg in all cases as well mass spectrometry coupling can present specific difficulties. The use of various HILIC columns represents a good alternative as well. However, in our experience some of the target analytes only achieved higher LOQs than 0.01 mg/kg.
A critical comparison of the obtained results by the different approaches together with a robust extraction are critically evaluated.

O-04

Cationic polar pesticides in food and beverage matrices by IC-MS/MS

Alexander Semyonov, Terri Christison, John Madden

Thermo Fisher Scientific, 1214 Oakmead Parkway, Sunnyvale, CA 94088, USA

Recent developments in the analysis of anionic polar pesticides have led to an increase in testing and regulation in surface and drinking water as well as food and beverages samples. However, developments in the analysis of cationic polar pesticides have lagged their anionic counterparts, primarily because of the analytical challenges. Advances using the Quick Polar Pesticides (QuPPe) method combined with IC-MS/MS have successfully demonstrated the applicability for the analysis of anionic polar pesticides, particularly glyphosate. However, for cationic polar pesticides, previous work has demonstrated an inability to resolve the Diquat / Paraquat peak pair. This combined with their similar m/z ratios makes it difficult to use IC-MS/MS methods for this application. Cationic polar pesticides such as chlormequat, diquat, mepiquat and paraquat may occur as residues in food, but may not be included in pesticide monitoring programs due to the difficulty in the determination of these target analytes using generic multi-residue methods.
To address this analytical challenge, we have recently developed the Thermo Scientific™ Dionex™ IonPac™ CS21-Fast-4µm column to provide satisfactory separation of the four key quaternary polar pesticides: chlormequat, diquat, mepiquat and paraquat. This work demonstrates a new IC-MS/MS method to identify and quantify low concentrations of quaternary amine cationic polar pesticides in foods and beverages IC-MS/MS. In this oral presentation, we will discuss the IC-MS/MS method and demonstrate its applicability for determination of chlormequat, diquat, mepiquat and paraquat in the extracts of food and beverage samples prepared using the QuPPe sample preparation technique.

O-05

Improving the QuEChERS Liquid/Liquid Extraction of Analytes with Widely Varying Physicochemical Properties: Example of 201 Veterinary Drugs in Milk

Anton Kaufmann,[1] Mirjam Widmer[1],Kathryn Maden[1], Patrick Butcher[1],and Stephan Walker;[1]

[1]**Official Food Control Authority of the Canton of Zürich,** Fehrenstrasse 15; Zürich, 8032, Switzerland; anton.kaufmann@kl.zh.ch;

A novel liquid/liquid extraction and clean-up process for the multiresidue analysis of veterinary drugs in milk is described. The paper addresses limitations of existing QuEChERS based protocols. This refers to poor and irreproducible recoveries of frequently used drugs (e.g. penicillins, tetracyclines and quinolones). Replacing the conventional QuEChERS salts by a mixture of di-potassium hydrogen phosphate and potassium dihydrogen phosphate not only improves critical analyte recoveries but also significantly reduces the amount of inorganic salts dissolved in the acetonitrile phase.
The methodology produces quantitative data and has been fully validated according to the current EU validation guidelines by utilizing two different high resolution mass spectrometry instruments coupled to liquid chromatography (LC-Q-Orbitrap and LC-Q-TOF). The multiresidue method covers virtually all milk matrix relevant veterinary drugs (except nitrofurans and aminoglycosides). The simplicity and absence of expensive consumables makes it attractive for high-throughput routine analysis.

O-06

Assessment of a new cartridge design in automated miniaturized solid-phase extraction cleanup for pesticides and environmental contaminants in fatty and nonfatty matrices

Nicolás Michlig and Steven J. Lehotay

USDA Agricultural Research Service, 600 East Mermaid Lane; Wyndmoor, PA 19038, USA; nicolas.michlig@usda.gov

Instrument-top sample preparation (ITSP), also known as µSPE, has been used for several years in QuEChERS (and now QuEChERSER) for automated cleanup in parallel with (low-pressure)GC-MS(/MS) analysis of pesticides and environmental contaminants in fatty and nonfatty foods. The ITSP cartridges contain 20 mg anh. $MgSO_4$, 12 mg of primary secondary amine (PSA), 12 mg C18, and 1 mg CarbonX sorbents to retain water, fatty acids, lipids, and chlorophyll, respectively. The cleanup entails robotic autosampler loading of 300 µL of initial extract (after the salting out step) into the cartridges at 2 µL/s, from which ≈220 µL of final extract elutes into the receiving microvials housed within autosampler vials for immediate injection in (LP)GC-MS(/MS). Excellent selectivity (cleanup), trueness, and precision have been demonstrated for a wide scope of analytes (pesticides, PCBs, PAHs, and PBDEs), particularly when using internal standards. Recently, µSPE cartridges with a new design were introduced that contain the same sorbents (except CarbonX is replaced with graphitized carbon black) packed into a 1-piece polypropylene housing. Rather than relying on friction between the syringe needle and septum cap as in ITSP, the new µSPE cartridges are gripped by applying pressure to the needle along the tapered opening tube. This also forms a seal to allow leak-free operation at higher flow rates than ITSP. A study was conducted to evaluate and re-optimize µSPE conditions in QuEChERSER for up to 265 diverse analytes in hemp pellets, spinach, whole milk, avocado, lamb muscle, and eggs. The results demonstrated that 500 µL loading volume at 5 µL/s gave excellent cleanup and 80-120% recoveries for 90-96% of the analytes with RSDs typically <5%. The dead volumes were reduced from ≈80 to ≈50 µL and automated reliability with the new cartridges was improved compared to the ITSP design.

O-07

The Study of Ultra-high Performance Liquid Chromatography Electrospray Ionization Q-Orbitrap Mass Spectrometry and Various Extraction Methods for Fingerprinting and Identification of Molecular Authenticity Markers from Apple Juices and Grape Juices

Jian Wang[1], Willis Chow[1]

[1] **Canadian Food Inspection Agency**, Calgary Laboratory, 3650-36th Street N.W., Calgary, Alberta, T2L 2L1, Canada

This study explored the use of ultra-high performance liquid chromatography electrospray ionization quadrupole Orbitrap high resolution mass spectrometry (UHPLC/ESI Q-Orbitrap MS) and three extraction methods that included Dilution (1:1 with solvent buffer), QuEChERS Step I and QuEChERS Step I&II for fingerprinting and identification of molecular authenticity markers in apple juices and grape juices. Of the three extraction methods, Dilution allowed for the most number of compounds to be detected for fingerprinting and revealed the most differences between samples. However, the three methods yielded similar PCA (principle component analysis) score plot pattern recognition (clusters, relative positioning). Dilution can be used for fingerprinting and profiling of apple juices and grape juices but QuEChERS Step I was preferred because it provided relatively clean sample extracts, which helped to maintain the performance of Q-Orbitrap over time for routine applications. QuEChERS Step I provided reproducible PCA score plots and extracted key markers such as kaempferol 3-O-glucoside and quercetin 3-O-glucoside (flavonals), phloridzin (phloretin 2'-O-glucoside; chalcones), and resveratrol and piceid (polydatin; stilbenes, non-flavonoid phenols) from apple juices and grape juices. Resveratrol, piceid, kaempferol 3-O-glucoside, and quercetin 3-O-glucoside were in relatively high amount in red grape juices while phloridzin was in high amount in apple juices. These markers often showed similar patterns (amounts) in the trend charts, and their amounts varied among the same type of juice. The UHPLC/ESI Q-Orbitrap along with the Dilution or QuEChERS Step I methods can serve as a practical HRMS-based non-target metabolomics approach for fingerprinting and identification of markers, which can be potentially used to develop quantitative methods, for juice authentication.

O-08

Analysis of Aminoglycosides in Foods using LC-MS/MS with a Zwitterionic Stationary Phase Column

Jinchuan Yang and Paul Rainville

Waters Corporation, 34 Maple Street, Milford MA 01757, USA; Jinchuan_yang@waters.com

Aminoglycosides (AMGs) are an important class of antibiotics that have been extensively used in both human and veterinary medicine. Extra-label use of AMGs in animal husbandry can lead to high residue level of AMGs in foods. The presence of AMGs in food represents a risk to consumer health due to their toxicity, allergenicity, and possible contribution to antimicrobial resistance. It is important to monitor AMG content in food to ensure the proper use of AMGs and the safety of food.
In this study, the Atlantis™ Premier BEH™ Z-HILIC Column, which has a zwitterionic sulfoalkylbetaine stationary phase on BEH particles, was evaluated for the analysis of seventeen AMGs in food by electrospray tandem mass spectrometry (ESI-MS/MS). The AMGs included in this study were amikacin, apramycin, gentamicin (C1, C1a, C2/C2a), hygromycin B, kanamycin, kasugamycin, neomycin, neamine, paromomycin, ribostamycin, spectinomycin, streptomycin, dihydrostreptomycin, sisomicin, and tobramycin. The effects of chromatographic conditions, such as mobile phases, pH, and ionic strength, on the separation of seventeen AMGs were systematically investigated. Gradient elution with a binary mobile phase of aqueous 20 mM ammonium formate at pH 3.0 and acetonitrile with 0.1% formic acid provided a reliable and adequate separation with excellent sensitivity for these AMGs. The extraction of food samples was carried out using a trichloroacetic acid containing solution. A solid phase extraction (SPE) and clean-up procedure was optimized using an Oasis™ HLB Cartridge. This method was evaluated for milk, beef, pork, liver, and honey samples. Performance characteristics for sensitivity, accuracy, and precision will be presented.

O-09

Bringing reliable determination of anionic polar pesticides in food to the routine laboratory

Simon Hird,[1] Dimple Shah,[2] Benjamin Wuyts,[3] and Stuart Adams;[1]

[1]**Water Corporation**, Wilmslow, SK9 4AX, UK; simon_hird@waters.com; [2]Waters Corporation, Milford, MA 01757, USA, [3]Waters Corporation, 2600 Antwerp, Belgium

Interest in the determination of highly polar, anionic pesticides in agricultural produce and foodstuffs has noticeably increased over the last ten years, driven by the potential safety concerns around glyphosate, and other reported residues. As a result, routine testing labs have strived to achieve efficient and reliable methodology to meet the demands of increased surveillance and brand protection. Methods were needed that could be used to determine residues of a series of highly polar, anionic pesticides and relevant metabolites, in a range of commodities, without resorting to derivatization or ion pair reagents. We evaluated various chromatographic options, focusing on the attributes of retention, separation, sensitivity, and reliability. The Torus DEA column, originally designed for supercritical fluid chromatography, is packed with ethylene bridged hybrid (BEH) particles with a diethylamine (DEA) bonded phase. It was chosen as it was anticipated it would provide a combination of hydrophilic interaction liquid chromatography (HILIC) and weak anion exchange (WAX) attributes using mobile phases compatible with mass spectrometry (MS). The column was shown to be capable of analysis of a wide range of highly polar, anionic pesticides in a single analytical run. Further optimization of conditions followed to improve sensitivity culminating with the launch of a new Anionic Polar Pesticides Column, based upon the same BEH DEA particles but packed in an UPLC format column. This presentation will focus on the lessons learned during the initial evaluations, the development of the Torus DEA method, the new the Anionic Polar Pesticide column and recent changes to methodology.

O-10

The use of honey as an environmental biomonitor of contamination in Natural Park of Montesinho: a comparison of extraction methods for the determination of multiple contaminants

Virgínia Cruz Fernandes[1], Diana Rede[1], Mónica Oliveira[2], and Cristina Delerue-Matos[1]

[1]**REQUIMTE/LAQV, Instituto Superior de Engenharia, Instituto Politécnico do Porto,** Rua Drº António Bernardino de Almeida, 431, 4200-072 Porto Portugal; vircru@gmail.com/virginiacruz@graq.isep.ipp.pt;

[2]Escola Superior de Saúde, Instituto Politécnico do Porto**,** Rua Drº António Bernardino de Almeida, 400, 4200-072 Porto Portugal;

The recent decline of honeybee colonies is a problem of global concern. While there are multiple variables, including poor nutrition, diseases, and loss of natural bee habitat, negatively affecting bee health, it is becoming increasingly clear that the widespread use of pesticides on crops and other environmental contaminants is a major factor. During honey production, bees collect nectar from different plants in the surrounding area of their hives, and the deposition and/or absorption of environmental pollutants (through the air, soil, and water) might influence the honey's quality. Therefore, honey could represent a bioindicator of environmental quality. As such, to preserve honeybee health, which is inextricably integrated with human health, and to preserve the quality of bee by-products, especially honey, requires regular monitoring using rigorous analytical methods to confirm product quality and safety.
This study aims to compare different extraction approaches. Honey was extracted by QuEChERS and/or dispersive solid-phase extraction and/or ultrasonic extraction (US). The methods were evaluated regarding the accuracy, precision, recoveries, matrix effects, and versatility. The results proved that US coupled with one step QuEChERS was the most efficient method for the simultaneous extraction of multiple contaminants in honey. In this study, we used GC-ECD/GC-MS instruments for the identification/quantification of pesticides, flame retardants and polychlorinated biphenyls. In a quantitative validation, acceptable performances were achieved with overall recoveries 70-120% and <20% RSD for >25 analytes in >20 sample extracts at 3 spiking levels. After analytical validation, the methodology was applied in honey samples from Natural Park of Montesinho in Portugal.

O-11

A Robust High-Throughput GC/MS/MS Analysis of 203 Pesticides in Under 10 Minutes with Helium and Hydrogen Carrier Gasses

Anastasia A. Andrianova[1], Eric Fausett[1], and Bruce D. Quimby[1]
[1] **Agilent Technologies**, 2850 Centerville Road, Wilmington DE 19808

There is a growing demand for rapid methods for the analysis of chemical residues. This work describes achieving fast GC/MS/MS analysis, while maintaining robust system performance in complex food matrices. Two system configurations using mid-column backflush provide robust analysis in 10 minutes while maintaining sufficient chromatographic resolution for the analysis of 203 compounds. A conventional 15m x 15m (0.25mm x 0.25µm) column configuration was used with helium carrier gas and a narrow bore 10m x 10m (0.18mm x 0.18µm) was used with helium and hydrogen carrier gasses.
The conventional 20-min retention time-locked method for 203 pesticides was used as a benchmark for the optimized fast analyses. To achieve faster analysis, two approaches were taken with helium carrier gas. First, the same 15m x 15m conventional mid-column backflush configuration was used with a faster oven ramp, yielding the analysis time of 10 min. Second, a narrow bore 10m x 10m mid-column backflush configuration was used enabling 10-min analysis time. The latter method was precisely scaled using method translation. In addition, the narrow bore column 10m x 10m was used with hydrogen carrier gas enabling 8-min analysis time.
A novel EI source was used for pesticides analysis with hydrogen carrier gas. The new source is optimized to address undesired in-source hydrogen-related issues like hydrogenation and provided improved performance with hydrogen carrier gas in chemical residue analysis with GC/MS.
The two presented column configurations enable fast and robust chromatographic analysis of 203 pesticides in under 10 minutes with helium and hydrogen carrier gases.

O-12

Simultaneous Determination of Alternaria Toxins, Ergot Alkaloid Epimers, and Other Major Mycotoxins in Various Food Matrices by LC-MS/MS

Shun-Hsin Liang, Jamie York, Joe Konschnik, Hansjoerg Majer, and Justin Steimling

Restek Corporation, 110 Benner Circle; Bellefonte, PA 16823, USA; Shun-HsinLiang@restek.com

Various food commodities are vulnerable to different types of fungal pathogens and could be contaminated with differential classes of mycotoxins as a result. It is ideally to implement a generic method for simultaneous determination of multi-mycotoxins in different food matrices or agricultural products. In this study, a simplified sample preparation procedure and a reliable LC-MS/MS analytical method was developed for comprehensive measurement of 37 regulated and emerging mycotoxins including 5 *Alternaria* toxins, 6 major ergot alkaloids and their corresponding epimers.
Four different food matrices (baby wheat cereal, peanut, tomato puree, and blended flour) were chosen for method validation to demonstrate the applicability of this analytical method to a wide range of food types. Sample extraction was performed using a formic acid-acidified 80:20 acetonitrile:water solution followed by extract dry-down and reconstitution in a 50:50 water:methanol solution for injection analysis on a Biphenyl LC column. Chromatographic analysis was performed using MS-friendly acidic mobile phases and completed with a short 11-minute cycling time for proper separation of ergot alkaloid epimers. Method accuracy and precision was evaluated by fortification of food samples at 3 different levels. Accurate quantification was achieved using matrix-matched calibration standards at the range of 0.4 to 400 µg/kg. The recoveries of all mycotoxins (except citrinin) in fortified samples were from 70% to 120%, and the relative standard deviation was less than 20%. The established workflow was simple and fast for multi-mycotoxin determination with a unique benefit of simultaneous analysis of *Alternaria* toxins and ergot alkaloids.

O-13

Robust Analysis of Pesticide Residues in Black Tea by Improved GC-MS/MS

Carlos Parra, Jerry Mueller, Jonathan DeCenzi, Katarzyna Banaszewski

NOW Health Group, 395 S Glen Ellyn Rd; Bloomingdale, IL 60108, USA; carlos.parra@nowfoods.com

With the growing emphasis on wellness and customer focus on prevention rather than treatment of misalignments, the interest in dietary supplements rapidly increased in recent years. The global market is projected to reach $278 billion in 2024 and just in the United States, dietary supplements are used by more than 80% of adults. The increased use of dietary supplements raises public health concerns about their safety and efficacy both, short and long term. Many manufacturers strive to remain compliant and provide their customers with high quality product by using fit for purpose methods. Botanical matrices, such as black tea, are very challenging when it comes to pesticide residue analysis due to their complexity. To overcome the challenges and to ensure the safety of dietary ingredients used in a manufacturing process, sensitive and robust instrumentation is needed in addition to an appropriate sample preparation method. A study was conducted to evaluate the performance of a new TRACE 1610 GC paired with TSQ 9610 MS/MS, when analyzing 200+ pesticide residues in black tea. All monitored analytes met the SANTE guidelines and offered significantly improved sensitivity, when compared with a previous generation GC-MS/MS. Additionally, the change in the hardware design improved uptime and allows to perform maintenance without interrupting the workflow, thus maximizes the lab productivity.

O-14

Development and Application of a Multiresidue Method for the Analysis of Pesticides in Plasma

Michael S. Gross[1] and Michelle L Hladik[1]

[1]**U.S. Geological Survey**, California Water Science Center, 6000 J St.; Sacramento, CA 95819, USA; msgross@usgs.gov

Nontarget organisms are exposed to pesticides following their applications in agricultural and urban settings, potentially resulting in deleterious effects. Direct measurements of pesticides in biological tissues may better characterize exposure and possible toxicological endpoints versus analyses in environmental media (*i.e.*, water, soil, and air). Plasma represents a nonlethal sample medium that can be used to assess recent exposures to contaminants. Herein, a method is developed and applied to quantify nearly 200 pesticides and their metabolites or degradates in small volume plasma samples (≤ 250 µL). Plasma samples are protein precipitated with a 3.5:1 ratio of 0.5% formic acid in acetonitrile to sample. Pass-through solid phase extraction is used to clean-up samples and remove lipids. Samples are analyzed by liquid chromatography and gas chromatography tandem mass spectrometry. Recoveries of pesticides, metabolites, or degradates ranged from 51 to 150%. The developed method was applied to bird, fish, and manatee plasma to show proof of application. Multiresidue methods for pesticides in biological media help improve understanding of the fate of bioactive compounds in the environment and of the exposure of nontarget organisms.

O-15

What ISO 17034 Accreditation of Reference Materials Mean for Your Quality Assurance/Quality Control Program

Daniel R. Biggerstaff

LGC Standards, 7290B Investment Drive, North Charleston, SC 29418, USA; dan.biggerstaff@lgcgroup.com;

A large portion of any laboratory's Quality Assurance/Quality Control (QA/QC) program relies on the validity of the analytical data generated. The importance of not only having accurate reference materials for analytical instrument calibration and quality control but also understanding the Total Combined Uncertainty of those reference materials cannot be overstated. Most reference materials now provide an uncertainty value on the Certificate of Analysis. It is important, however, to understand which aspects of the production and life of the reference material are being included. ISO 17034 was written specifically for reference material manufacturers to ensure that the standards they produce are accurate and have well defined uncertainties. The differences between Reference Materials and Certified Reference Materials will be discussed along with the different aspects of Total Combined Uncertainty. The importance of neat material characterization and traceability, production techniques, human error, reference material verification, and four different components that contribute to the total combined uncertainty will be discussed. Reducing human error in the preparation of daily working standards will also be discussed. ISO 17034 accreditation provides a way for the analytical testing laboratory to have confidence in the data it is providing.

O-16

The Principle of Pooled Calibrations Indicates Shrinking the Valid Range of pH Measurements

Jens E.T. Andersen, Mercy Menong

Department of Chemical and Forensic Sciences, Faculty of Sciences, Botswana International University of Science and Technology, Boseja Ward, Private Bag 016, Palapye, Botswana, andersenj@biust.ac.bw

Despite the simplicity of pH measurement, evaluation of the uncertainty as a function of the pH values appears to be far from simple with respect to both calculations and understanding. The measurand may be defined as the proton interaction with the surface of the glass electrode but it is not defined as the pH value itself. According to the principle of pooled calibrations, all data were retained, no outliers were rejected, and no uncertainty budget was required for the method validation. Glass electrodes of two vendors were employed in the analysis and the pH ranges were given as -2 to 16 and 0 -14 for the two pH meters. The pH meters were calibrated by using certified buffer solutions at 25°C and the measurements of standards and samples were also performed at this temperature. The uncertainty of the standards complied with those of the samples, but the overall level of uncertainty reached values of U(pH) = 0.26 within the pH range of approximately 3 – 11. Outside this range of pH values, the uncertainty was considerably larger, and it was suggested to not trust pH values lower than 1 because of U(pH) = 1.4. In terms of hydronium concentration, the lower limit of analysis (LLA) was estimated as 10^{-14} molal for one of the pH meters and 10^{-11} molal for the other pH meter. The corresponding upper limit of analysis (ULA) was determined as 10^{-1} molal for both pH meters. These figures of merits may be used for routine pH measurements.

O-17

Challenges in Determining Acceptability of Dietary Supplement Raw Materials Based on Pesticide Residue Results Obtained Using In-House Methodology

Jonathan DeCenzi, Katarzyna Banaszewski

NOW Health Group, 244 Knollwood Dr; Bloomingdale, IL 60108, USA; jonathan.decenzi@nowfoods.com

Majority of dietary supplement raw materials do not have established tolerances for pesticide residues listed in 40 CFR 180, CODEX Alimentarius, or other international standards that may be referenced when manufacturing goods intended for global commerce. The enforcement of zero-tolerance Maximum Residue Limits (MRLs) en masse is not practical or evidence-based, while consumer safety and product quality is the driving force behind the industry investment in developing, validating and implementing in-house pesticide residue analytical methods. USP <561> offers some guidance regarding acceptable pesticide residue limits in dietary supplements, although this general chapter represents a fraction of the residues tested for in a typical screening panel. Additionally, some of the limits set forth in USP <561> would be considered unacceptable to some consumers of dietary supplements and could potentially be flagged as non-compliant in a foreign country. The reality is, manufacturers are often faced with difficult decisions regarding the acceptability of materials intended for distribution as dietary supplements. Risk analysis in making these decisions would be improved with harmonization amongst global regulations and clearer guidance in handling method uncertainty, variation between lab results and matrix interferences. This assessment explores incorporating these variables, as well as a dose-dependent toxicological approach to determining the acceptability of dietary supplement ingredients.

O-19

Determination of mycotoxins in dried fruits using LC-MS – homogeneity, troubleshooting, and confirmation of identity

Kai Zhang,[1] Steven Tan,[1,2] David Xu,[1,2]

[1]**US Food and Drug Administration,** Center for Food Safety and Applied Nutrition, Office of Regulatory Science, HFS-717. 5001 Campus Drive, College Park, MD, 20740; kai.zhang@fda.hhs.gov; [2]University of Maryland, Joint Institute for Food Safety and Applied Nutrition, 2134 Patapsco Building, 5145 Campus Drive, College Park, MD 20740

The FDA has been regulating and monitoring mycotoxins since the 1960s. Recently, a multi-mycotoxin LC-MS method was developed to further improve the agency's ability to detect a wide range of mycotoxins. To monitor co-exposure to toxic mycotoxins in foods, it is advantageous to simultaneously determine multiple mycotoxins using a single extraction and stable isotope dilution LC-MS analysis. In this matrix extension study, we applied a previously validated stable isotope dilution and LC-MS multi-mycotoxin method for analysis of dried fruits such as raisins, plums, figs, and cranberries. Samples were prepared using cryogenic grinding, fortified with carbon-13 (^{13}C) uniformly labeled internal standards for 12 target mycotoxins, and then extracted using 50% acetonitrile. Homogeneity of prepared samples was achieved using cryogenic grinding and was defined as particle size Dv90 <850 µm for the tested matrices characterized using a laser diffraction particle size analyzer but not conventional sieving procedures. The majority of spike recoveries in the four matrices for aflatoxins and ochratoxin A at 1, 10, and 100 ng/g; fumonisins, T-2 toxin, HT-2 toxin, and zearalenone at 10, 100, and 1,000 ng/g, ranged from 80 to 120% with relative standard deviations of <20%. Deoxynivalenol was not detected at 10 and 100 ng/g in plums. Additional troubleshooting procedures using liquid-liquid extraction, solid phase extraction, and elution gradient were evaluated to improve the detectability of the mycotoxin. Furthermore, we confirmed the identity of detected mycotoxins, ochratoxin A and deoxynivalenol, in incurred samples using enhanced product ion scans and spectral library matching.

O-20

Validation of QuEChERSER for PFAS in Food with Method and Instrumental Comparisons

Raegyn B. Taylor, Yelena Sapozhnikova

USDA Agricultural Research Service, 600 East Mermaid Lane; Wyndmoor, PA 19038, USA; raegyn.taylor@usda.gov

Per- and polyfluoroalkyl substances are wide-spread, persistent environmental contaminants that serve many industrial purposes. The strong carbon-fluorine bond allows for excellent stability in consumer applications, but this also leads to limited degradation and thus environmental contamination, which has ultimately led to several reports of contaminated food. Despite longer-chain PFAS being banned in several countries due to widespread occurrence and accumulation, they continue to be detected alongside emerging alternatives. Thus, monitoring food for PFAS continues to be imperative to understanding human exposure. This study (1) evaluated the relatively new QuEChERSER method against current sample preparation techniques for PFAS analysis in food (2) performed method validation in six USDA FSIS-regulated foods, and (3) compared analysis on high-resolution and triple-quadrupole mass spectrometers. Results include a robust comparison of extraction efficiency, lipid cleanup, and matrix effects for 33 targeted PFAS in beef, catfish, and eggs. QuEChERSER performed exceptionally well in comparison to other methods, so it was subsequently validated in beef, catfish, chicken, pork, liquid eggs, and powdered eggs. Recoveries were within 70 and 120% for all matrices at 1 and 5 ng/g food and for most analytes at the 0.1 ng/g level using a HRMS method. Matrix effects were less than 20% for all but one analyte. Analysis by triple-quadrupole MS provided better recoveries at the 0.1 ng/g spiking level but was outperformed by HRMS at higher levels. The validated method provides an easy extraction approach to quantifying PFAS in food, with an analysis method allowing for concurrent nontarget screening of samples.

O-21

Pesticides Exposure Assessment: Analytical Method Challenges and Opportunities

Amos K. Dwamena, Ph.D.,

Smithers Environmental Risk Sciences, Wareham, MA; USA

Pesticides, including both traditional plant protection products and biocides, although produced under very strict regulatory guidelines to have minimal impact on human health and environment, may prompt testing requirements under FIFRA for human health impacts of workers exposed to pesticide products. Exposure to pesticides can occur through multiple routes including oral, dermal, and inhalation depending on the type and use of the material. The direct (point-of-contact) and indirect (scenario exposure) measurement of pesticides offer accurate techniques to measure exposure to contaminants in the breathing zone, in food and drink, and on the skin. Developing robust analytical extraction methods with unconventional matrices for monitoring whole body dosimetry such as outer clothing, inner clothing, and gloves, dermal exposure through gauze pads, as well as inhalation exposure through glass fiber filters or OVS tubes is critical to generate reliable data to model post-application risks. The analytical challenges involving sample preparation, sample sizes, and extraction encountered with supporting worker exposure studies would be discussed.

O-22

Measuring Wild Bee Exposure to Pesticides Using Silicone Bands and Tissue Residues

Michelle L. Hladik,[1] Johanna M. Kraus,[2] Kelly L. Smalling,[3]

[1]**USGS California Water Science Center**, 6000 J St, Placer Hall; Sacramento, CA 95819, USA; mhladik@usgs.gov; [2]USGS Columbia Environmental Research Center; Columbia, MO 65201, USA; [3]USGS New Jersey Water Science Center; Lawrenceville, NJ 08648, USA

Wild bees are important pollinators of agricultural crops, but there are limited data on their field-level pesticide exposures. It can be difficult to collect enough wild bees to achieve the desired sample mass for analysis, and there may be other techniques that can serve as a proxy for wild bee pesticide exposures. To determine if silicone bands used as passive samplers (to capture aerial application/drift) could serve as an acceptable proxy for bee exposure, pesticides were measured in silicone bands and bee tissue from fields in Iowa. The sites were grasslands within an agricultural landscape (corn and soybeans as the dominant crops) and were measured twice, July and August, to represent different parts of the growing season. The bands were placed in the fields for one month prior to retrieval, which coincided with bee sample collection. Out of the 180 pesticides and degradates analyzed (using both liquid and gas chromatography with tandem mass spectrometry), 49 compounds were detected: 11 herbicides/degradates, 20 insecticides/degradates, 17 fungicides, and a plant growth regulator. Of the pesticides detected, 20 were observed in both silicone bands and bees, 19 in just the bands, and 10 in only the bee tissue. Of the 10 pesticides detected only in the bees, five were degradates/metabolites indicating the potential for the bees to transform the parent pesticide, and all were detected infrequently (<5% of samples). Results from this study indicate that silicone bands can provide a cheap, effective, and non-lethal method to measure bee exposure to pesticides.

O-23

PFAS Analytical Method Development and Troubleshooting

Charles R. Powley

Center for PFAS Solutions, 272 Quigley Blvd; New Castle, DE 19720, USA; chuck.powley@pfasolutions.org

Per- and Polyfluoroalkyl Substances (PFAS) are known as “forever chemicals that are everywhere”, and analytical capabilities are needed to quantify single digit part per trillion levels in water and part per billion levels in all other environmental and biological matrices. Published and even certified methods are available, but even with these the challenges facing an analytical laboratory wishing to implement them are daunting. Background detections are common and can be attributed to laboratory contamination and PFAS impurities in sample processing equipment and high purity solvents used for the HPLC-MS/MS determinations. False positives can arise even after standard contamination prevention measures are undertaken and it is best to follow a protocol for tracking down the source using the general practice of working backwards through the method to isolate the step(s) where the contamination is introduced and then drilling down to identify the specific issue. Some examples of non-obvious sources of contamination are pipet tips and solid phase extraction (SPE) cartridges containing PFAS compounds, either as impurities or added deliberately. Other challenges with PFAS methods arise from the non-stick nature of these compounds, so SPE concentration and purification steps may fail in certain cases. Also, as the number of PFAS compounds added to methods increases, potential problems with volatility and even stability can arise. Once again, if poor precision and/or accuracy data are obtained, it is best to work backwards through the method and track down the specific cause(s).

O-24

Chemistry, Chromatography & Cannabis: Troubleshooting the Cannabis Workflow for Analysis

Patricia Atkins
Spex, an Antylia Scientific Company, 203 Norcross Rd, Metuchen, NJ 08840; patricia.atkins@antylia.com

Cannabis testing has become a popular focus for analytical testing discussions and regulation. While standardization organizations and governmental agencies continue to work to fill the previous vacuum of information, test methods and regulatory guidelines laboratories struggle with the analysis workflow and the difficulties that come with cannabis analyses.
The analytical workflow has three general steps including sample preparation, sample processing and sample analysis. At each step of the workflow there are opportunities to increase accuracy and efficiency, and an equal number of chances to introduce error. This presentation will break down the theories behind each step of the process and give best practice suggestions to reduce error and contamination to increase accuracy.
Sample preparation will look at the different methods of sample commutation and the effect of sample homogeneity and sample reduction conditions on process efficiency. Sample processing and analysis will look at the chemistry of the different target compounds and the principles of chromatography in how they apply to cannabis method development.

O-25

Pesticide Residue Testing: Challenges Faced with Routine Analysis

Sarah M. King,[1] Cheryl Stephenson,[1] John Reuther[1]

[1]**Eurofins Central Analytical Laboratory, 2219 Lakeshore Drive; New Orleans, LA 70122**

Pesticide testing laboratories are confronted with many challenges. One of the largest challenges is keeping up with the wide array of matrices submitted for testing. Analysis of new matrices can usually be achieved by employing the laboratories' routine methods; however, that is not always the case. This presentation will discuss the obstacles that occur with challenging matrices and what strategies are employed to resolve these difficulties.

O-26

Evaluation of coated blade extractions prior to LC-HRMS for the analysis of veterinary drug residues in aquaculture products

Jessica P. Rafson, Sherri B. Turnipseed, and Mark R. Madson

Animal Drugs Research Center, Office of Regulatory Affairs, U.S. Food and Drug Administration, Denver Federal Center, Denver, CO 80225, USA; Jessica.Rafson@fda.hhs.gov

Solid-phase microextraction (SPME) has been demonstrated for a wide range of analytes, including veterinary drug residues. Although not an exhaustive extraction technique, SPME can be used for trace-level, quantitative analyses when coupled with mass spectrometry. Some advantages of using SPME are the avoidance of organic solvents, lower matrix effects than exhaustive solvent extractions, and simplified extraction procedures. There are many commercially available SPME geometries, but one of the latest to become available are coated blades which have a biocompatible coating embedded with adsorbent particles for extraction. This work focuses on evaluating coated blade extractions for veterinary drug residues in aquaculture samples. A wide variety of veterinary drug residues representing 16 compound

classes were spiked at their screening target testing levels into fish samples which were then extracted using coated blades prior to solvent desorption and analysis using an established LC-HRMS method. Before optimization, 79-84% of the 90 veterinary drug residues spiked in salmon, shrimp, and tilapia could be detected at low part-per-billion levels. Changing time, temperature, and agitation had a modest effect on compound retrieval, but the desorption solvent had a large impact on which compounds could be desorbed and analyzed. Initial desorption optimization experiments used spiked aqueous samples to simplify extraction before using a complex aquaculture matrix. Additionally, matrix effects were low when using coated blades (average ion suppression ~10% compared to solvent standards). Investigations into method optimizations to improve sensitivity are continuing.

O-27

Development of a confirmatory method for the detection of gestagens in animal liver using a simplified extraction with LC-MS and high-field asymmetric waveform ion mobility spectrometry (FAIMS)

Randy W. Purves,[1,2] Ratnadip Vaghela,[1] Jana Kinar,[1] Bryn O. Shurmer [1]

[1]Canadian Food Inspection Agency, 116 Veterinary Road, Saskatoon, Canada S7N 0X4; randy.purves@inspection.gc.ca;
[2]University of Saskatchewan, 51 Campus Dr, Saskatoon, Canada S7N 5A8;

Gestagens are a class of veterinary drugs that are used to promote growth in animals slaughtered for food. Gestagens are banned in the EU and some other countries, but are approved for use in other jurisdictions. Canada, the USA and the Codex Alimentarius Commission have established maximum residue limits (MRL) for melengestrol acetate (MGA); in Canada, MRL values are 0.014 ppm in fat and 0.006 ppm in liver. At the CFIA, we have been developing a confirmatory method for three gestagens in animal liver, most importantly MGA, but also megestrol acetate (MA), and chlormadinone acetate (CA). The protocol in fat currently used at the CFIA is time consuming and was developed several years ago. For liver, a simplified extraction procedure using acetonitrile with magnesium and sodium salts, and no clean-up steps, was investigated. The procedure also employed an isotopically labelled internal standard, d3-MGA. When using liquid chromatography mass spectrometry (LC-MS), an elevated chemical background was observed, which was most notable near the desired detection limits (0.6 ppb). Differential ion mobility techniques separate gas-phase ions and we implemented a high-field asymmetric waveform ion mobility spectrometry (FAIMS) interface in combination with LC-MS to reduce chemical background. Optimum values of transmission through the FAIMS interface were established and at these values, transmission of the chemical background was greatly minimized. When using the same conditions, signal-to-noise (S/N) values were improved roughly 10-fold with LC-FAIMS-MS compared with LC-SRM for the three gestagens. Analytical performance and results from method validation will be presented.

O-28

Optimization of multi-residues screening method for the detection of antibiotics in Milk products: the case of Labneh.

Ghinwa Ismail [1 2], Khaled El Hawari[2], Farouk Jaber[2], Eric Verdon[1]

[1] Anses, French National (NRL) and EU Reference Laboratory (EU-RL) for Veterinary Medicinal Product and Antimicrobial Residues in Food from Animal Origin, Laboratory of Fougères; Fougères; France
[2] CNRSL, Lebanese Atomic Energy Commission, Laboratory for Analysis of Organic Compounds; Lebanon

Corresponding Authors: ghinwa.isma.ext@anses.fr / ghinwa.ismail0@gmail.com / khaled.hawari@cnrs.edu.lb

Antibiotics can be administered in food-producing animals for therapeutic purposes. Inappropriate administration or disrespect of withdrawal time lead to the presence of residues in animal tissues and their fluids, such as milk. This can

cause several undesirable effects for consumers like allergies, intestinal flora perturbation linked to antibacterial resistance, carcinogenic disruption and inhibition of lactic bacteria growth during yogurt manufacturing. Therefore, regulations worldwide have been enforced to set up their maximum residue level in milk and animal products. These regulations apply to the primary product like raw milk and should be relevant for manufactured dairy products as well.
Labneh is a dairy product highly consumed in Lebanon. It is a semi solid composition prepared through removal of water and water-soluble compounds present in yogurt by straining. This product is slightly acidic and contains 6.42 %-10.70% lipids, 8.24-10.43 % proteins and 2.86-4.91% lactose.
Because there is no such surveillance on the antibiotic residues in milk and dairy products in Lebanon, a screening method was developed for the detection of antimicrobial residues in dairy products, and to evaluate the influence of the manufacturing process on the evolution of contamination.
In this study, we optimize a multi-residue LC-MS/MS screening method based on liquid-liquidextraction using acetonitrile and a simple purification step, to analyze 74 antibiotics in 30 Labneh samples collected from different regions in Lebanon. In addition, milk used for the preparation of some Labneh samples is also analyzed to determine the effect of the manufacturing on the degree of contamination.

O-29

Evaluation of the uncertainty in differing approaches to correct for matrix effects in LC-MS/MS analysis of antibiotics and growth promoters in fish products

Ryan Gibbs, Laura Albrecht, Janelle Samson, Jason Smith, Gina Benedict

Canadian Food Inspection Agency, 1992 Agency Drive; Dartmouth, NS B3B 1Y9, Canada; Ryan.Gibbs@inspection.gc.ca

Measurement uncertainty (MU) is an important performance indicator of analytical methods for the determination of residues. There are two general approaches to determine the MU of a method: the “bottom-up” approach, as recommended by the Bureau International des Poids et Measures in their “Guide to the Expression of Uncertainty in Measurement”, where each individual source of uncertainty in a protocol is combined to determine the MU; and the “top-down” approach, as described by Barwick and Ellison, in which the results of method validation studies or routine quality control (QC) data can be used to make an assessment of MU. When using the top-down approach, it is possible to overlook the uncertainty associated with the differences between samples since validation and QC data often use repeated measurements of the same sample. The European Union has recently released new performance guidance (CIR/2021/808) requiring the evaluation of “relative matrix effect” where the instrument response differences between at least 20 different sample lots is required when evaluating matrix effects (ME). A variety of techniques to correct for ME have been described, such as internal standardization, standard addition and recovery correction. The MU differences in each approach were evaluated in a multi-class method for the analysis of antibiotics and growth promoters. Each approach offers advantages and disadvantages in terms of costs, workload, and MU. The results of each approach and the choice of approach based on risk factors and client needs will be discussed.

O-30

Low Level LC-MS/MS Determination of Cannabinoids in Isolates, Hemp Plant Materials, and Extraction By-Products

Lukas Vaclavik, John Schmitz, Michael Buhrman, Vanessa Snyder, Grace Bandong

Eurofins Food and Chemistry Testing, 6304 Ronald Reagan Avenue, Madison, WI 53704, USA; lukasvaclavik@eurofins.co.uk

In the last few years, the popularity of hemp and hemp-based products has significantly increased. Commercial consumer products with cannabidiol (CBD) and other cannabinoids, such as dietary supplements, cosmetics or vape liquids have become subject of high demand. These items are usually formulated with the use of high purity CBD isolates obtained through hemp plant extraction. In addition to CBD, other minor cannabinoids can be co-isolated during the production process and carry through the purification process, including compounds with hallucinogenic effects (e.g. tetrahydrocannabinol, cannabinol, etc.) which are subject to regulation in many countries worldwide. Additionally, hemp plant, hemp seeds, seed oil and their processing by-products may represent valuable assets to livestock and poultry industries as feedstuff. Considering the above, reliable and sensitive methods are required for analysis of hallucinogenic and other cannabinoids in these matrices. In this study, a liquid chromatography-tandem mass spectrometry (LC-MS/MS) method was developed and validated for quantification of fifteen cannabinoids including Δ^9- and Δ^8-tetrahydrocannabinol (THC), tetrahydrocannabinolic acid (THCA), tetrahydrocannabivarin (THCV), and cannabinol (CBN) in CBD isolates, hemp seeds, meal and protein matrices. Sample preparation and chromatographic separation were based on AOAC Official Method of Analysis AOAC 2018.11. The method was thoroughly validated to demonstrate adequate performance at a target limit of quantification (LOQ) of 1 mg/kg. Acceptable accuracy and precision with recoveries within 70-120% and RSDs ≤20% was achieved. The presented method was shown to be suitable for routine quantification of relevant cannabinoids at low levels in wide range of matrices.

O-31

The Pros and Cons of Mass Spectrometry for Cannabis Potency Testing

Anthony Macherone

Agilent Technologies, 2850 Centerville RD; Wilmington, DE 19808, USA; anthony_macherone@agilent.com

The “gold standard” for determining potency in cannabis and hemp is HPLC with UV detection. Although UV offers some molecular information, it is generally considered a non-selective detector like FID with GC systems. Mass spectrometry (MS) is highly selective and more sensitive that UV or FID although sensitivity may not be a concern with cannabis or hemp matrices. This is because the US DEA threshold of 0.3% (wt./wt.) that distinguishes cannabis from hemp equals 3 mg/g or 3000 ppm and dilution of the sample is required to get it into calibration ranges on the order of µg/mL. Therefore, the power of MS is selectivity but even this is not always the case. Consider Δ9-tetrahydrocannabinoil (THC) and cannabidiol (CBD). These two cannabinoids are structurally different but share the same empirical formula and the same quasi-molecular ion in electrospray, so their identification still relies on retention time. THC and CBD can however be distinguished from interferences at their respective retention times with MS which is generally not possible with UV or FID.

Given the complexity of cannabis matrices and the and the increasing occurrences of novel cannabinoids like cannabichromene, THC-acetate, Δ10-THC, cannabinoid diastereomers and enantiomers, etc., we suggest MS is a superior methodology for regulatory potency testing. This presentation will discuss the pros and cons of MS and tandem quadrupole MS with liquid and gas chromatography and compare these to UV and FID. We will highlight the power of MS and contrast this with potential pitfalls to consider before switching to MS.

O-32

Results from two CannaQAP Interlaboratory Study Exercises at NIST for Improving Cannabinoid, Toxic Element, and Moisture Measurements in the Cannabis Industry

Walter Brent Wilson,[1]

[1]**National Institute of Standards and Technology**, 100 Bureau Dr; Gaithersburg, MD 20889, USA; walter.wilson@nist.gov;

A *Cannabis* Quality Assurance Program (CannaQAP) was recently established to help improve the comparability of the analytical measurements across the cannabis industry for both hemp and marijuana. CannaQAP is an interlaboratory study mechanism that is similar to a proficiency testing scheme; however, the focus is towards education without assigning pass/fail grades to anonymized participants. All CannaQAP studies are evaluated by NIST, participants are provided with a preliminary report/certificates, and summarized in publicly available NIST Internal Reports. Results from these studies will help inform NIST of current challenges to assist in the design and characterization of cannabis reference materials (RMs). Exercise 2 of CannaQAP focused on the determination of cannabinoids (Δ^9-THC, THCA, total THC, CBD, CBDA, etc.), toxic elements (As, Cd, Pb, Hg, etc.), and moisture in six cannabis samples. Similar studies were included in Exercise 3 and two cannabis oils were included for cannabinoid study. In this presentation, the results from both exercises of CannaQAP will be summarized to evaluate the current status of these measurements in the cannabis industry. In addition, future plans for CannaQAP will be included for the expansion of the analytes (pesticides, terpenes, etc.) and sample matrix (edibles, vape liquids, etc.).

O-33

Navigating Unchartered Territory: Cannabis Testing Landscape from an Honest Perspective.

Katherine M. Evans. Longboard Scientific Consulting Corporation. Colorado Springs, CO; USA; Kate.Evans@longboardscientific.com

Entering the cannabis laboratory testing space can be an exciting endeavor for entrepreneurs and scientists alike. However, there are many complexities to consider from regulations, lack of standardization, recruiting talent, competition, cost of analysis just as examples. This discussion will touch on and present a real-world view of some of the challenges that cannabis testing presents particularly testing methods and best practices that are being adapted from other industries. Before jumping all into this type of project- there needs to be a solid framework – such as a business and financial plan, timelines particularly focused on the work involved for validation of test methods.

As quoted by Deepak Chopra: **"**All great changes are preceded by chaos." Thankfully there is a trend in the industry to improve and overcome some of the obvious mistakes and blunders made over the last several years. The goal of this presentation is to provide an overview of some observed practices and possible solutions/fixes and where the cannabis testing industry is heading both short and long term.

O-34

A novel LC/MS/MS method with ESI and APCI ion source for analysis of 102 pesticides and 5 mycotoxins regulated by Colorado state in Hemp

Avinash Dalmia[1],

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

A Novel LC/MS/MS method with dual ESI and APCI source was developed for analysis of 102 pesticides and 5 mycotoxins regulated by Colorado state in hemp. We would demonstrate how we measured non-polar pesticides (normally analyzed by GC/MS/MS) such as quintozene, chlordane, etridiazole, fenvalerate, iprodione, chlorfenapyr, methyl parathion, endosulfan and others using LC/MS/MS. The ionization mechanism of non-polar pesticides using APCI ion source would be elucidated. Hemp causes matrix effects and interference. We carried out MS and LC method optimization and changed ion source from ESI to APCI for challenging analytes to mitigate hemp matrix and interference effects to achieve lower detection limits. Automated solvent extraction was used for sample preparation since it is quicker, simpler, cheaper, reproducible and greener way to achieve extraction efficiency of 80-120%. We added 30 internal standards to compensate for hemp matrix effects in order to achieve method accuracy of 70-120%.

POSTER ABSTRACTS

P-01

Mechanistic insights into phenanthrene acropetal translocation via wheat xylem: separation and identification of transfer proteins

Nengde Zeng,[1,2] Yuting Zhu,[1] Dongru Wang,[1] Ruonan Chen,[1] Qiurun Feng,[1] Xinhua Zhan,[1] Baoshan Xing;[2]

[1]**College of Resources and Environmental Sciences**, Nanjing Agricultural University; Nanjing, Jiangsu Province, 210095, People's Republic of China; 2020203020@stu.njau.edu.cn;[2]University of Massachusetts, Amherst, USA

Polycyclic aromatic hydrocarbons (PAHs) have the potential to cause cancer, teratogenicity, and mutagenesis in humans. Food chains have emerged as a critical pathway for PAH exposure in humans. However, the acropetal transfer of PAHs in staple crop stems, particularly in xylem, a critical path, is unknown. We initially discovered that peroxidase (uniprot accession: A0A3B5XXD0) and unidentified proteins with a deep hydrophobic ligand-binding pocket may function as carriers to load and acropetally translocate phenanthrene (a model PAH) in wheat xylem. These two proteins are the key factors in the solubilization of phenanthrene in wheat xylem sap. The magnetic phenanthrene-bound bead immunoassay confirmed that the two proteins bind to phenanthrene selectively. Protein structural studies and molecular docking revealed that the protein-phenanthrene complex may form via hydrophobic interactions in the conservative binding region. Under phenanthrene exposure, a substantial quantity of peroxidase was produced, as well as an unusually high expression of uncharacterized proteins, which aided in the acropetal transfer of phenanthrene in wheat xylem. Our findings provide fresh light on the molecular mechanism of PAH loading in plant xylem, as well as techniques for ensuring the security of staple crops and improving the efficacy of phytoremediation in a PAH-contaminated environment.

P-02

Development of validated GC-MS/MS based confirmatory method for dioxin-like POPs in stack air and bottom ash matrices – An affordable approach for assessing emissions in developing countries

Ajay S.V.[1,2], Kirankumar P.S. [1,2], Prathish K.P.[1,2]
[1]**Environmental Technology Division, CSIR- National Institute for Interdisciplinary Science and Technology**, (CSIR-NIIST), Thiruvananthapuram, Kerala, India -695 019
[2] Academy of Scientific and Innovative Research (AcSIR), Ghaziabad-201002, India

Uncontrolled combustion of solid wastes is identified as the largest source of dioxins-like persistent organic pollutants (dl-POPs-PCDD/F/PCBs) emission in developing countries. Dl-POPs are notified class-A carcinogens and a methodical accounting of its emissions at source level is critical for inventorisation and to understand the cumulative health risk. Low cost, innovative solutions are particularly relevant for developing nations due to limited analytical facilities and resources, which puts a huge population under risk from environmental and food contaminants. In this study a GC-MS/MS based method was developed for the analysis of dl-POPs in environmental matrices and validated with respect to method limit of quantification, trueness, intermediate precision, experiments at maximum level and control of recoveries. A customized open burn test facility was constructed to carry out simulated solid waste combustion studies, as well as enabled quantitative sampling of air and residue emissions. The samples were analyzed using the validated method and estimated the national default emission factor for dl-POPs from open burning of MSW as 180 µgTEQ/ton. The annual emission in India was calculated to be 13.4 kgTEQ, which is quite high considering the low tolerable monthly intake for humans - 70 pgTEQ/kg/bw. Further, onsite ambient air emission studies were conducted to estimate the carcinogenic and non-carcinogenic health risk to the exposed community as per ATSDR guidelines. The considerable risk observed at the study sites to the human population were reported which could lead to effective policy decisions on waste management thereby ensuring environmental and food safety.

P-03

Correlation of PCDD/Fs and PCBs levels between fish and sediment samples and health risk assessment to consumers: A prima facie study carried out in an industrial hotspot located in south India by validated GC MS-MS analytical protocol

P. S. Kirankumar[1], S. V. Ajay[1], Amala Varghese[1], K. P. Prathish[1]

[1]CSIR-National Institute for Interdisciplinary Science and Technology, Thiruvananthapuram, Kerala, India; prathishkp@niist.res.in

A cost effective alternative to HRGC-Magnetic sector HRMS remains a bottleneck in enabling routine monitoring of highly toxic dioxins and PCBs in food and environmental samples. The present study depicts the development of an affordable GC MS/MS based methodology and its validation in accordance with EU 644/2017 to quantify PCDD/Fs and PCBs in fish and sediment matrices. The critical parameters such as limit of quantification (LOQ), spike recovery at ML, ML/2 and 2 ML levels, internal standard recoveries and measurement uncertainties were estimated. A novel protocol based on lowest acceptable calibration point which exhibited RRF (<30%), RSD of response factors (±15%), relative ion intensity (≤15%) was employed, to attain matrix specific limit of quantification. The spike recovery tests at ML demonstrated the trueness, precision and measurement uncertainty and assessed its compliance with the regulation. The developed analytical methodology was employed to quantify PCDD/Fs and PCBs in fish and sediment samples collected from an industrial hotspot located in south India for the first time. In most of the analysed fish samples the levels of dioxins and PCBs were below the EU maximum level except species like *M. cephalus* and *E. surattensis which were* collected near to the industrial area. The statistically significant correlation analysis showed strong positive correlation of pollutants between fish and sediment samples, which indicates the need for regulating sediment levels. Moreover, the health risk assessment to the fish consumers denoted moderate to high health risk to adults in the case of *M. cephalus* and *E. surattensis* samples.

P-04

Persistence of two Pyrethroid Insecticide Residues under Greenhouse Conditions

George Antonious, Kentucky State University

Permethrin and cypermethrin are two insecticides commonly used in agricultural production systems in Kentucky and worldwide. They are very similar in chemical structure, but cypermethrin contains a cyanide (CN) group. The objectives of this investigation were to: 1) Separate permethrin and cypermethrin residues using a single extraction and purification procedure. 2) Determine the dissipation constants and $T_{1/2}$ values of permethrin and cypermethrin residues on bean and cucumber leaves and fruits grown under greenhouse conditions. 3) Determine fruit harvest time with respect to the maximum residue limits (MRLs) and greenhouse worker's re-entry intervals. Residues were monitored using a gas chromatograph (GC) equipped with an electron capture detector (GC-ECD) and GC equipped with a Mass Selective Detector (MSD). Residues of the two insecticides revealed the presence of permethrin isomers at retention times of 26 and 26.6 min that correspond to the cis and trans-isomers, respectively. The GC also revealed the presence of four cypermethrin isomers at retention times of 30.3, 30.9, 31.3, and 31.5 min. The average initial deposits of permethrin were 2.7 and 0.2 on cucumber leaves and fruits surfaces, respectively. Whereas cypermethrin initial deposits were 5 and 2 µg g-1 on cucumber leaves and fruits, respectively indicating greater deposits on leaves than on fruits. $T_{1/2}$ values of permethrin and cypermethrin residues on beans pods (7.2 and 9.5 d, respectively) and cucumber fruits (13 and 3.3 d), respectively indicated a waiting period of 10 and 15 d are required for consumption of cucumber fruits and bean pods sprayed with cypermethrin to drop the residues to the maximum allowable limits.

P-05

Estimation of Heavy Metals and Fluoride Ion (F^-) in Vegetables Grown Nearby the Stretch of River Yamuna, Delhi (NCR), India

Sadre Alam,[1] Laxmi Kant Bhardwaj,[2] Rwitabrata Mallick,[1] Swapnil Rai[1]

1-Department of Environmental Science, Amity University Madhya Pradesh
2- Amity Institute of Environmental Sciences (AIES), Amity University, Noida (India); bhardwaj.laxmikant@gmail.com

Heavy metals are very hazardous to humans and the environment. They are non-biodegradable and can enter in humans through contaminated water and food. After entering, they are deposited in fat tissues, bones and can cause multi-organ failure. The aim of this study was to investigate the concentration of Pb, Cu, Cd, Hg, Cr, As and F^- in the vegetables which were grown near the stretch of Yamuna river, Delhi (NCR). A total of 32 vegetable samples were collected from Mayur Vihar, Near Kalindi Kunj, and Near Thermal Power plant in three different seasons (2017-2019). In the monsoon season, the highest concentration of Pb, Cu, Cd, Cr were found 18.05 ppm in spinach, 32.60 ppm in lady finger, 2.59 ppm in radish, 6.60 ppm in lady finger. In the summer seasons, Pb, Cu, Cd, Cr were found 1.58 ppm in spinach & radish, 2.65 ppm in radish, 0.32 ppm in radish, 0.25 ppm in methi. In the winter seasons, Pb, Cu, Cr were found 17.08 ppm in radish, 2.84 ppm in beet, 4.39 ppm in spinach. The highest concentration of F^- was found 4.35 ppm in radish. Hg and As were found BDL in all different vegetable samples in all seasons. This study concludes that all three sites were found to be contaminated with the concentration of heavy metals.

P-06

Determination of 8α-hydroxymutilin as a marker residue for tiamulin in swine tissue by liquid chromatography–tandem mass spectrometry

Shizuka Saito-Shida, Nao Kashiwabara, Satoru Nemoto, Hiroshi Akiyama

National Institute of Health Sciences, Tonomachi 3-25-26, Kawasaki-ku, Kawasaki, Kanagawa 210-9501, Japan; shizsaito@nihs.go.jp

Tiamulin is a semi-synthetic derivative of the natural antibiotic pleuromutilin and is widely used as a veterinary drug for swine. Herein, we report the development of a sensitive and reliable method for determining 8α-hydroxymutilin as a marker residue for tiamulin in swine tissue using liquid chromatography–tandem mass spectrometry (LC-MS/MS). The method consists of sample extraction with acetone, defatting by acetonitrile/hexane partitioning, hydrolysis of the tiamulin metabolites to 8α-hydroxymutilin under alkaline conditions, liquid-liquid extraction with ethyl acetate, cleanup using a primary secondary amine cartridge, and LC-MS/MS analysis. The developed method was validated for 8α-hydroxymutilin in swine muscle, fat, and liver at two levels, namely 0.01 mg/kg and the maximum residue limits established in Japan (i.e., 0.1 mg/kg for swine muscle and fat, and 0.6 mg/kg for liver). The trueness ranged from 82 to 89%, and the relative standard deviations ranged from 1 to 3%. No chromatographic interference was observed near the retention time of 8α-hydroxymutilin, and matrix effects were negligible for all matrices, suggesting that the cleanup protocol was effective. The calibration curve was linear in the 0.005–0.5 µg/mL range, with a coefficient of determination greater than 0.997. The developed method enabled accurate quantification using solvent-based calibration without compensating for matrix effects and losses during sample preparation. The limit of detection of the method was 0.0005 mg/kg for each matrix. The developed method is suitable for regulatory-purpose analysis of 8α-hydroxymutilin as a marker residue for tiamulin as defined by the European Union and several other countries.

P-08

Determination of β-Agonists in Animal Tissues by LC-MS/MS

Wendy Y, Zeyu H, Gloria W.

R&D Dept. MERIEUX NutriSciences, Silliker JR Lab. Burnaby,Canada

Since the residues of beta-agonists present in meat products can be a risk to the health of consumers, the use of beta-agonists as growth promoters has been banned in most countries. Consequently, it is important to monitor the abuse of these compounds. This study aimed at the determination of total β-agonists in animal tissues. Homogenized animal tissues were enzymatically hydrolyzed for 16 to 18 hours at 37±1 °C with β-glucuronidase in the buffer solution of sodium acetate. Subsequently, the β-agonists and their glucuronide conjugates were extracted from the sample matrix using acetonitrile. The extract was cleaned up by EMR-Lipid dSPE and the samples were analyzed using API 6500 plus MS/MS coupled with an Agilent 1260 Infinity II LC system. The detection limit is 0.1 ng/g for Ractopamine and Zilpaterol; 0.5 ng/g for Brombuterol, Cimaterol, Clenbuterol, Clenpenterol, Hydroxymethyl clenbuterol, Isoxsuprine, Mabuterol, Ritodrine, Salbutamol, Terbutaline and Tulobuterol; 1 ng/g for Mapenterol, Clenproperol, Formoterol and Metaproterenol.

P-09

Evaluation of a Mycotoxin in Animal Feed Candidate Reference Material

Fabrizio Straccia[1,2], Jacolin A. Murray[1]

[1]**National Institute of Standards & Technology, Chemical Sciences Division**, 100 Bureau Drive, MS 8392; Gaithersburg, MD, USA; jacolin.murray@nist.gov ; [2] **Instituto Nacional de Tecnología Industrial,** (INTI) Buenos Aires, Argentina; fstraccia@inti.gob.ar

Corn Distiller's dried grains with solubles (DDGS) is a common by-product of ethanol production. Due to its nutritional value, DDGS is an excellent low-cost animal feed ingredient that is produced in large quantities.

Fungi of the genera Aspergillus, Fusarium, and Penicillium produce mycotoxins such as Aflatoxins, Fumonisin, Deoxynivalenol, Zearalenone, Ochratoxin A, HT-2 toxin, T-2 toxin. Like many grain-based ingredients, DDGS may contain mycotoxins such as Fumonisin, Deoxynivalenol and Zearalenone that can originate from the grains used to produce the DDGS, especially if stored under warm or humid conditions. Mycotoxins are not destroyed in the production process of DDGS and can negatively affect animal health and/or be transferred to the human food chain. Due to the increased production and use of DDGS as feed and the increasing needs of laboratories moving toward LC-MS based multi-mycotoxin analysis, the National Institute of Standards and Technology (NIST) and National Institute of industrial technology (INTI) are collaborating to produce a naturally contaminated reference material (RM) for mycotoxins in DDGS. Bulk DDGS candidate material was obtained and is currently being evaluated. Evaluation of the material includes LC-MS/MS analytical method development, screening the candidate RM for mycotoxins, investigating the impact of particle size distribution on mycotoxins extraction efficiencies and homogeneity, and the impact of irradiation of the material. These initial studies will determine if the number and levels of mycotoxins in the material are appropriate to produce a fit-for-purpose RM and if the bulk material needs further processing (milling, irradiation, etc.) prior to packaging.

P-10

Development of Comprehensive Multi-class veterinary drug mixtures for food testing

Jens Seltmann[1]

[1]**LGC Standards**, Bgm.-Schlosser-Str. 6A; 86199 Augsburg, Germany; jens.seltmann@lgcgroup.com;

Residues from veterinary drugs are a well-known threat to human health. A variety of these compounds are banned for use in food for human consumption and many more are regulated with specific maximum residue limits. As the industry moves to multi-residue screening methods it is imperative that laboratories can conduct analyses of veterinary drugs accurately and efficiently. This is particularly problematic due to the complex nature of veterinary compounds and Reference Materials play a crucial role in mitigating these issues. In this poster we will present the development process for six multi-component mixtures containing 92 analytes of different classes of vet drugs including Tetracyclines, Sulfonamides, Avermectins, Quinolones, Benzimidazoles and Beta-lactams to support analytical labs screening for these compounds. The stability of all mixtures was intensively investigated through real-time stability monitoring over several years. We will also be exploring the extension of these mixtures including the development of pharmaceutical mixtures for environmental testing.

P-11

Pyrrolizidine Alkaloid Contamination in Plant-based Dietary Supplements and Food Matrices

Michael Buhrman, John Schmitz, and Lukas Vaclavik

Eurofins Food Chemistry Testing, 6304 Ronald Reagan Avenue, Madison, WI 53704, USA; michaelbuhrman@eurofinsus.com

Pyrrolizidine alkaloids (PA) are a group of natural toxins synthesized within several plant species that are ubiquitous in nature. From previous scientific studies, many PAs have been identified as contaminants in specific foodstuffs, including tea, honey, dried herbs, and herbal dietary supplements. Considering PA are genotoxic carcinogens to humans, maximum levels have been set by the European Commission in Regulation 2020/2040 for a number of commodities. Working from the current European Union (EU) regulation, there is a pressing need for the evaluation of these matrices with increased sensitivity and specificity to allow for the accurate quantification of PA and reporting of the summed PA that are detected. In this study, we performed a thorough validation of a LC-MS/MS method based on BfR (German Federal Institute for Risk Assessment) assay in a range of matrices to cover and expand upon the Commission Regulation 2020/2040 in the evaluation of pyrrolizidine alkaloids and their N-oxides. Matrices included tea, honey, dried herbs including hemp plant, spices, seeds, milk products, and various forms of herbal dietary supplements. Validation data will be presented for a range of matrices evaluated at levels meant to represent a contamination level consistent with meeting the reporting requirements dictated by the EU legislation.

P-12

Is sewage sludge a source of priority emerging micropollutants for agricultural soils?

Diana Rede, Ivan Teixeira, Marta Oliveira, Cristina Delerue-Matos, and **Virgínia da Cruz Fernandes**;

REQUIMTE/LAQV, Instituto Superior de Engenharia do Porto, Instituto Politécnico do Porto, Rua Dr. António Bernardino de Almeida, 431, 4200-072 Porto, Portugal; virginiacruz@graq.isep.ipp.pt;

Crop intensification reduces both soil productivity and biodiversity, and without proper fertility, the agricultural ecosystem cannot function. Sewage sludge (SS) is worldwide reused in agriculture as an environmentally friendly alternative to synthetic fertilizers. Thus, the identification and quantification of pollutants in SS requires the development of selective methods to prevent ecological risks. The aim of the present work was to develop a Quick, Easy, Cheap, Effective, Rugged and Safe (QuEChERS) extraction method, coupled with chromatography techniques to quantify 43 organic compounds from different chemical families (i.e., organophosphorus, organochlorine and pyrethroid pesticides, organophosphates esters flame retardants, polybrominated diphenyl ethers, polychlorinated biphenyl compounds, and polycyclic aromatic hydrocarbons) in stabilized SS samples. The analytical method was validated through the evaluation of the recoveries, the reproducibility, and the limits of detection and quantification. The recoveries ranged between 50–126% and the reproducibility of the method was ≤21% expressed as relative standard deviation. The method allows the analysis of the target compounds with limits of detection ranging from 0.007 and 0.271 μg g^{-1}, and limits of quantification ranging from 0.024 and 0.821 μg g^{-1}. The procedure was applied to 7 distinct SS samples, collected in Wastewater Treatment Plants from the northern of Portugal. The results showed the presence of 16 target compounds with concentrations ranging from 0.042 to 26.80 μg g^{-1} (dw). Eight of the detected compounds are considered persistent organic pollutants, and one of them, α-endosulfan I, was detected in all samples with concentrations between 0.110 and 0.571 μg g^{-1} (dw).

P-13

The Pesticide Data Program 1991-2021: 30 Years of Residue Data and Trends

Brenda Foos,[1] Amy Gaines,[1] Jessica Murillo,[1] and Chris Pappas,[2]

[1]**USDA Agricultural Marketing Service**, 1400 Independence Avenue, SW, Washington, DC 20250, USA Brenda.Foos@usda.gov
[2]Retired, **USDA Agricultural Marketing Service**, 1400 Independence Avenue, SW, Washington, DC 20250, USA

The Pesticide Data Program (PDP) entered its 30th year in 2021 as one of the world's largest monitoring programs for pesticide residues. Administered by the U.S. Department of Agriculture (USDA) Agricultural Marketing Service (AMS), PDP is a Federal-State partnership and tests a wide variety of domestic and imported foods using a statistically sound sampling program and standardized laboratory methods. The PDP database contains over 42 million data points for pesticide residue-commodity pairs resulting from the analysis of nearly 310,000 food samples of 126 different commodities. The average residues reported per sample has increased from 1 in 1991 to 271 in 2020. Data trends have been reviewed for four example commodities that have been analyzed multiple times throughout the course of the program's history. The results illustrate that pesticide residues on foods change over time, particularly when new pesticides are registered, and are typically below the maximum allowable pesticide residue limits established for food in the US. Updated data, such as those provided by PDP, are important for pesticide exposure and risk assessment as well as international trade.

P-14

Influence of preparation conditions over the homogeneity uncertainty of in-house Quality Control Samples of LC-MS/MS amenable pesticide residues in food matrices

Andres S. Salinas, Laura V. Morales, Yeraldine Aguilar, Ivonne A. Gonzalez, and Gina A. Torres;

National Metrology Institute–INM of Colombia, Ak 50 #26-55 int. 2, Bogotá, Colombia; assalinas@inm.gov.co;

In-house Quality Control Samples (QCM) are an alternative to the limited supply of reference materials for method validation and assurance of the validity of pesticide residue results. These are prepared within the facilities in which they will be used, and must meet homogeneity and stability criteria to be considered adequate to demonstrate that a measurement system is under statistical control. In this work, the effect of the preparation conditions of QCMs fortified with pesticide residues in banana and rice on the uncertainty due to homogeneity was studied. Candidates for multi-pesticide QCM in rice and banana were prepared following the guidelines of ISO Guide 80:2014. For both matrices, the effect of the composition of the spiking solvent on the uncertainty due to homogeneity was studied. In addition, the effect of the drying method (oven and freeze-drying) was studied for rice, while for banana only freeze-drying was used. The uncertainty homogeneity between units was evaluated following a method based on QuEChERS and measurement by LC-MS/MS through a completely randomized block design and statistical analysis by two-way ANOVA. For rice and banana, uncertainties for homogeneity of the order of 0.1-0.7% and 0.1-3.0%, respectively, were obtained when using a predominantly aqueous solvent (>70%) as a medium for spiking, compared to uncertainties up to 50 times higher for several analytes when using an organic medium (>70%). Regarding the effect of the drying method for rice, appropriate uncertainties were obtained with both, provided that a predominantly aqueous solvent is used.

P-15

Monitoring pesticides and other contaminants in plants surrounding beehives: a case study of Natural Park of Montesinho

Virgínia Cruz Fernandes, Diana Rede, Inês Casal, and Cristina Delerue-Matos

[1]**REQUIMTE/LAQV, Instituto Superior de Engenharia, Instituto Politécnico do Porto,** Rua Drº António Bernardino de Almeida, 431, 4200-072 Porto Portugal; vircru@gmail.com/virginiacruz@graq.isep.ipp.pt;

People, animals, and the environment can be exposed to multiple chemicals from a variety of sources. The co-occurrence, co-exposure, and potential combined effects (additive, synergistic or antagonistic) of multiple chemicals in the environment and consequently in the honeybees is a problem of global concern. Bees collect nectar from several plants in the surrounding area of their hives, and the deposition and/or absorption of pesticides and other environmental pollutants (through the air, soil, and water) might influence bee health. As such, to preserve bee health, which is inextricably integrated with human health, and to preserve the quality of bee by-products, requires regular monitoring using rigorous analytical methods.
This study aims to compare different extraction approaches to monitor multiple contaminants from plants collected near beehives. Plants were extracted by QuEChERS and/or dispersive solid-phase extraction and/or ultrasonic extraction (US). The methods were evaluated regarding linearity, precision, recoveries, and versatility. In this study, we used GC-ECD for the identification/quantification of halogenated pesticides, brominated flame retardants and polychlorinated biphenyls. In a quantitative validation, acceptable performances were achieved with overall recoveries 72-120% and <20% RSD for > 30 analytes and LOD (7-17µg/L). After analytical validation, the methodology was applied in 40 plant samples from the Natural Park of Montesinho in Portugal.

P-16

The Detection of Flavonoids in Hemp Flower by LC-MS/MS

Jamie L. York

Restek Corporation, 110 Benner Circle; Bellefonte, PA 16823, USA; Jamie.york@restek.com

Flavonoids are a class of compounds that are endogenous in hemp and cannabis that are antioxidant rich and can affect the flavor profile. In this work, a method to detect 19 flavonoids in CBD and CBG dominant hemp flower was developed using LC-MS/MS. 500 mg of ground sample was weighed into a 50 mL centrifuge tube. 10 mL of methanol:water (80:20) was added prior to vortexing (5 seconds) and sonicating (15 minutes). The sample was then centrifuged for 5 minutes and the supernatant was diluted 50-fold in water:methanol (60:40), vortexed, and filtered using a 0.2 µm filter vial prior to analysis. The analytical method was developed using LC-MS/MS with electrospray ionization in positive and negative ion modes. The method uses gradient mobile phase conditions on a Raptor Biphenyl 100 x 2.1, 2.7 µm column equipped with a guard cartridge. The chromatographic method was able to resolve all isobars with an overall analysis time of 7 minutes. 10 flavonoids were detected in the CBG hemp flower sample and 12 flavonoids were detected in the CBD hemp flower sample. Single point recovery experiments were also performed at 100 ng/ml using two isotopically labeled internal standards. The results of the single point recovery ranged from 80 – 104%. The developed method was able to resolve isobars within a cycle time of 7 minutes and acceptable recoveries were achieved for both hemp flower samples, indicating this is an effective procedure for the extraction of flavonoids from hemp flower.

P-17

Analysis of Multiclass Multiresidue Pesticides in Complex Dry Spices Using the Enhanced Matrix Removal Passthrough Cleanup

Limian Zhao, Aimei Zou, and Anastasia Andrianova

Agilent Technologies Inc., 2850 Centerville Rd., Wilmington, DE, 19808

Dry spice samples are extremely challenging for pesticides analysis, due to their significant matrix complexity and extremely difficult to clean. The abundant matrix co-extractives cause problematic issues on both LC/MS/MS and GC/MS/MS and result in poor quantitation for targets. Current sample preparation methods either apply high dilution with compromise on method sensitivity, or use extensive sample cleanup but experience high risk of targets loss.

A simplified passthrough cleanup using the novel Enhanced Matrix Removal (EMR) cartridges was developed to provide an easy but efficient matrix cleanup after samples' QuEChERS extraction. The novel EMR cartridges adopt the use of an advanced hybrid carbon material, Carbon S sorbent, blended with other critical sorbents using the optimized formula and bed mass. Four typical dry seasoning samples were investigated, including cayenne pepper, black pepper, cumin and cinnamon. The method was developed and optimized by the thorough matrix removal study and over 200 pesticides quantitation using either LC/MS/MS or GC/MS/MS or both. Depending on the matrix complexity, a single EMR cartridge passthrough or two sequential EMR cartridges passthrough cleanup was developed. The sample preparation workflow demonstrated >50% of matrix removal, and >80% of targets with acceptable quantitation results. The method also demonstrated improved robustness on both LC/MS/MS and GC/MS/MS, contributed to less matrix accumulation on the flow path and MS source. Specifically, the sequential EMR passthrough cleanup was applied to the analysis of over 500 pesticides in black pepper by LC/MS/MS and demonstrated improved quantitation results over current methods.

P-18

Detection and quantitation of Per- and polyfluoroalkyl substances (PFAS) in pork meat using an LC-Orbitrap High-Resolution Mass Spectrometer

Ed George,[1] Maciej Bromirski[1]

[1]**Thermo Fisher Scientific**, 355 River Oaks Parkway, San Jose, CA 95134, USA

PFAS were first developed in the 1940s and have been used by industrial and commercial sectors for products that required thermal and chemical stability, water resistance, and stain resistance. Awareness of PFAS contamination in the environment first emerged in the late 1990s following developments in tandem LC-MS/MS instrumentation which enabled low-level target detection. Most regulations have been focused on environmental contamination of PFAS that have leached into water and soil samples from a variety of sources, such as landfills or Aqueous Film Forming Foam (AFFF) used to extinguish flammable liquid fires.

The need to analyze PFAS in other matrices is growing rapidly since they are very stable and readily bioaccumulate in plant and animal tissues. Moreover, there are over 9000 known PFAS (with more PFAS being actively discovered) and only a limited number of certified reference standards commercially available for routine targeted analysis. HRAM analysis by LC-Orbitrap has an inherent advantage over triple quadrupole MS because it can provide both quantification and identification of target PFAS, along with the option of retrospective analysis on samples that may contain untargeted PFAS. It can also overcome challenges of matrix interferences that have been observed in animal tissue extracts by tandem MS due to low ppm mass accuracy and high mass resolution capability of orbitrap instrumentation. This work describes an LC-HRAM method with excellent sensitivity and specificity demonstrated in pork meat, which is fit for purpose and has the potential to be an excellent platform for expanded PFAS analysis into more complex matrices.

P-19

Recent advancements in LC-MS/MS technology for enabling improvements in the multi-residue analysis of veterinary drugs in food

Ed George[1], Alan Atkins[3] , Laura Bruns[2], Dwayne Schrunk[2], Neloni Wijeratne[1], Claudia Martins[1]

[1] Thermo Fisher Scientific, San Jose, California [2] Iowa State University Diagnostic Laboratory, Department of Veterinary Diagnostic and Production Animal Medicine, College of Veterinary Medicine [3] Thermo Fisher Scientific, Hemel Hempstead, GB

Veterinary drugs are administered to animals to ensure animal welfare. It is necessary to screen food products for veterinary drug residues at the maximum residue limits (MRLs) set by global regulatory agencies. This screening typically involves identification and quantification of veterinary drugs using LC-MS/MS. Many publications and newer methodologies cite screening and quantification of large panels of multi-class veterinary drugs in a variety of matrices using a single or limited number of LC-MS/MS methods.

Here we describe an LC-MS/MS method for the quantitation of a diverse group of veterinary compounds spiked into pork muscle extract at concentrations equivalent to or below MRLs set in the China GB standard. Mobile phase composition, column, and liquid chromatography injection techniques were optimized to provide the best separation, peak shape, and response for the analytes studied. Importantly, the liquid chromatography flow path was evaluated prior to analysis with a system suitability mixture containing multiple classes of veterinary drug residues that elute over the retention time range of the method. The method takes full advantage of the Thermo Scientific™ TSQ Quantis™ Plus triple quadrupole mass spectrometer's advanced ion optics and fast polarity switching capability, which are critical for accurate quantification in high density SRM acquisition areas within the analytical run.

P-21

PestiMix - A quantum leap forward in multi-residue pesticide analysis

Kelly Cheshire

LGC Standards, 1 Chamberhall Business Park, Chamberhall Green, Bury, BL9 0AP, UK; Kelly.Cheshire@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. With this comes the inherent need to be able to accurately identify and quantify these contaminants with confidence while maintaining efficiency to deliver results on time. The development of multi-component Certified Reference Materials (CRM) plays a pivotal part in streamlining this process and ensuring measurement integrity. In this poster presentation we will discuss the latest developments of PestiMix which provides a quick and easy solution to enable prompt calibration and spiking of up to 745 pesticide analytes for multi-residue analysis via Liquid Chromatography and Gas Chromatography.

P-22

Sub 1 µg/kg detection of glyphosate and other anionic polar pesticides using QuPPe extraction and detection by LC-MS/MS

Dr Stuart J. Adams[1], Dr Gitte Barknowitz[1], **Dimple Shah[2] (dimple_shah@waters.com)**, Dr Kari L. Organtini[2]

[1] Waters Corporation, Wilmslow, UK, [2] Waters Corporation, Milford, USA

The area of anionic polar pesticide analysis has been evolving over the past 10 years with the adoption of generic extraction methods, such as the QuPPe[1] method, enabling laboratories to take a multi-residue approach for the analysis of these challenging analytes. The extraction performance of the QuPPe method is well documented and is a common approach for the extraction of polar pesticides from various food matrices. With the recent developments in MS detector technology, lower limits of detection and quantification can be achieved for this analysis by utilizing the enhanced negative ion sensitivity of the Xevo™ TQ Absolute Mass Spectrometer. This poster presents results from the performance of the LC-MS/MS method where limits of quantification of 0.5 µg/kg and 2 ug/kg in cereal samples are achievable. The trueness of the LC-MS/MS method was assessed over 10 injections at 1 and 10 µg/kg in cucumber matrix standards and at 10 and 50 µg/kg wheat flour matrix standards. Trueness in cucumber was between 91 to 117% with RSDs between 0.6 to 8.7% and between 96 to 104% in wheat flour with RSDs between 0.5 to 9.2%.

P-23

QuEChERS Extraction of Per-and Polyfluoroalkyl Substances (PFAS) from Edible Produce with Sensitive Analysis on Xevo™ TQ-XS Mass Spectrometer

Kari Organtini[1], Simon Hird[2], Stuart Adams[2], **Dimple Shah[1] (dimple_shah@waters.com)**

[1]**Waters Corporation**, 34 Maple St, Milford, USA
2Waters Corporation, Wilmslow, UK

The same sources of environmental per- and polyfluorinated alkyl substances (PFAS) exposure can also lead to contamination in food sources. Cultivating produce using PFAS contaminated water and soils can lead to the uptake of these compounds into the edible fruits and vegetables portions of plants. Thus, it is beneficial to have a straightforward method to monitor the occurrence of PFAS in produce. For this work, the FDA C-010.01 method based on the QuEChERS extraction method was implemented for extraction of PFAS using DisQuE dispersive solid phase extraction (dSPE) products followed by highly sensitive LC-MS/MS analysis on ACQUITY™ UPLC™ I-Class PLUS™ coupled to Xevo TQ-XS mass spectrometer. The method was evaluated in five different commodity types including lettuce, strawberry, cranberry, carrot, and potato. Some minor adjustments to the FDA procedure were included in this application to improve the chromatography for better quantitation and identification, and to improve extraction efficiency of target PFAS. These include a dilution prior to LC-MS/MS analysis to improve peak shape of early eluting analytes, removal of GCB to improve overall recovery, and use of buffered salts following AOAC protocol. A PFAS Kit was utilized to modify the LC to isolate possible system and solvent contaminants. This application for PFAS analysis in produce proved to be a simple, time efficient extraction, followed by an accurate, sensitive, and robust analysis for a range of 30 PFAS compounds of varying chemistry classes in the sub ng/g range.

P-24

The analyses of macrolides, tetracycline and sulfonamide antibiotics in animal tissues using LC-MS/MS

Simon Hird (simon_hird@waters.com)[1], Christelle Robert[2], Dmoulin Luc[2], Pierret Gilles[2], Natalie Gillard[2], Marijn Van Hulle[3]

[1]Waters coroporation, Wilmslow, UK
[2]CER Groupe, Marloie, Belgium
[3]Waters Corporation, Antwerp, Belgium

LC-MS/MS can be used to determine residues of the vet drugs of interest in a variety of matrices. Employing selective extraction and clean up options offers opportunities for the greatest sensitivity and robustness without relying on the highest specification instrument. This poster describes two methods for the determination of important classes of vet drug residues in some representative matrices. One deals with the determination of macrolides and another for the determination of a series of different antibiotics, namely tetracyclines, sulfonamides, trimethoprim, ormetoprim, and dapsone antibiotics. Both use solid-phase extraction (SPE) followed by LC-MS/MS using an ACQUITY™ UPLC™ I-Class PLUS™ System coupled to a Xevo™ TQ-S Micro MS/MS System. The methods both allow for a fast and reliable quantitation down to concentrations well below typical MRLs and have been successfully validated according to the European Commission Decision 2002/657. These cost-effective methods can be easily implemented in routine testing laboratories, has been demonstrated as suitable for checking compliance with MRLs, and has the potential for screening at much lower concentrations, such as for food business operators' due diligence testing.

P-25

Improved Sensitivity for the Detection of Per- and Polyfluorinated Alkyl Substances in Environmental Water Samples

Kari Organtini, Stuart Adams, Ken Rosnack, **Gordon Fujimoto** (Gordon_fujimoto@waters.com), Lindsay Hatch

Waters Corporation, 34 Maple St, Milford, MA 01757, USA; kari_organtini@waters.com

Sensitivity limits are constantly being challenged with the continued evolution in regulations and guidelines for per- and polyfluorinated alkyl substances (PFAS). Direct injection approaches have become popular for PFAS analysis since they reduce the sample preparation time, and more importantly limit the risk of introducing contamination. Generally, a large injection volume is needed to meet the ultra-low detection limits required. With the introduction of the enhanced sensitivity of a new tandem quadrupole, a wide range of PFAS can be detected in the ng/l range without employing a large volume. Water samples were diluted 1:1 in methanol, filtered then analyzed on a new high performance tandem quadrupole mass spectrometer with a compact design, enhanced negative ion detection and removable source shield to reduce source contamination from sample matrix and mobile phase buffer salts. PFAS evaluated included C4-C14 carboxylates and sulfonates, perfluoroethers (such as HFPO-DA or GenX), and precursors like telomer sulfonates. Initial evaluation was performed using solvent standards to understand instrument sensitivity. Peak area, peak height, and signal to noise were used to make this evaluation. Enhancements were experienced in all three parameters, indicating a true sensitivity increase without a detriment to signal to noise. Previous applications of the direct injection approach required a 30 µL injection volume, but with the increased sensitivity, injection volume was reduced to 10 µL. Even using the reduced injection volume, limit of detection (LOD), determined using a S:N of 3, for most compounds was near or below 1 ng/L.

P-26

Total Workflow for the Sensitive Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Fish, Meat, Edible Offal and Eggs

Kari Organtini[1], Stuart Adams[2], Simon Hird[2], **Gordon Fujimoto[1] (Gordon_fujimoto@waters.com)**

[1]Waters Corporation, 34 Maple Street, Milford, MA 01757, USA
[2]Waters Corporation, Wilmslow, UK

Rising concerns about the long-term impacts of human exposure to PFAS have propelled the scope of PFAS analysis from just environmental matrices into the field of food analysis as well. Over the last decade, cases of PFAS contamination being found in foods have become more prominent in the media. To protect the public and understand dietary exposure, analytical methods for the analysis of a large variety of food products are required. This study focused on methods for PFAS extraction and analysis in complex food samples of animal origin, which add complexity to sample preparation needs due to the presence of proteins and fats that can bind PFAS. For this study an alkaline extraction was performed using sodium hydroxide in methanol, followed by solid phase extraction (SPE) clean-up using mixed mode Weak Anion Exchange (WAX) chemistry with analysis performed using UHPLC-MS/MS. This extraction method was evaluated using a suite of 30 PFAS in six different food matrices: salmon, tilapia, ground beef, beef liver, beef kidney, and egg. Detection and quantitation limits were determined to be in the sub-ng/g range. Recoveries were within FDA criteria with utilization of isotope dilution for accurate correction of recovery during calculation of PFAS concentration in samples. Among the samples tested, five PFAS were detected in two different food samples purchased from local grocery stores. This comprehensive method allows for high confidence in results of PFAS in complex food matrices to allow for better monitoring and understanding of the environmental impact of PFAS on our food sources.

P-27

Development of a Multi-Toxin UPLC-MS/MS Method for 50 Mycotoxins and Tropane Alkaloids in Cereal Commodities

Nicola Dreolin[1], Henry Foddy[1], **Simon Hird (simon_hird@waters.com)**[1], Peter Hancock[1], Timothy Jenkin[1]

[1] **Waters Corporation, Wilmslow UK**

In this work we describe the performance of a multi-toxin UPLC™-MS/MS Method for 50 regulated and emerging mycotoxins, atropine, and scopolamine in cereal-based products. The high sensitivity tandem quadrupole mass spectrometer, the Xevo™ TQ-XS Mass spectrometer, was used in combination with an ACQUITY™ UPLC System to reach very low limits of detection and quantification. A mix of cereal flours was extracted using a simple “dilute - and-shoot” protocol, without any clean-up or internal standards. Calibration curves were plotted using solvent standards and spiked extract (matrix-matched calibration). Limits of detection and quantification were shown to be suitable for checking compliance with regulatory limits and to investigate the levels of other toxins. The lowest method limit of quantification (m-LOQ) was for aflatoxins (0.1 µg/kg). The calibration range was acceptable and covered three orders of magnitude for most analytes. Matrix effects were also calculated for all compounds and were found to be significant, illustrating the need for matrix-matched calibration. The method fulfilled the criteria set out in the SANTE guidelines for mycotoxins. Data was imported into the new waters_connect™ for quantitation and processed with the MS Quan app for an improved efficiency in data processing and review.

P-29

Method development and validation for the determination of pyrrolizidine alkaloids in a range of plant-based foods and honey using LC-MS/MS

Nicola Dreolin[1], Henry Foddy[1], Stuart Adams[1], Simon Hird[1], Peter Hancock[1], **Jeremy Shia (Jeremey_shia@waters.com)[2]**

[1] **Waters Corporation, Wilmslow UK**
[2] **Waters Corporation, 34 Maple Street, Milford, MA, USA**

Pyrrolizidine alkaloids (PAs) are toxins exclusively biosynthesised by plants. Expressing both genotoxic and carcinogenic properties, an increasing number of reports reveal relatively high contaminations with PAs in food, herbal infusions, and teas. This Application Note describes an analytical method for the determination of PAs in a variety of food commodities (tea, herbs, spices, cumin seeds and honey). Samples were extracted with a sulfuric acid solution, cleaned up using Oasis™ MCX SPE Cartridges, concentrated and resuspended prior to LC-MS/MS analysis. The chromatographic resolution of critical pairs of isomers was addressed in this study. Good method recoveries and excellent repeatability were obtained, which complied with the acceptance criteria in the CEN standard for single laboratory validation. Limits of quantification for individual compounds were 0.6 µg/kg, which were shown to exceed regulatory compliance, such that the method can also be applied to food intended for infants and young children.

P-30

Determination of pesticide residues in rice-based baby food using GC-MS/MS with APGC™ after extraction and clean up using QuEChERS

Dr Stuart J. Adams[1], Mrs Mette E. Poulsen[2], Mr Janathia De-Alwis[1], **Narendra Meruva (Narendra_meruva@waters.com)[3]**

[1]Waters Corporation, Wilmslow, UK, [2]National Food Institute, Technical University of Denmark, Lyngby, Denmark
[3] Waters Corporation, 34 Maple Street, Milford, MS, USA

Reliable analytical methods are needed for detection, quantification, and identification of hundreds of pesticide residues in many different commodities. This poster describes the development and validation of a comprehensive method based on GC–MS/MS for the determination of 166 pesticides in rice-based baby food. Extracts were prepared using a published version of QuEChERS for cereals followed by determination with GC-MS/MS. The use of GC-MS/MS utilizing atmospheric pressure ionization (APGC) has been shown to offer significant improvements in performance over electron ionization (EI) for pesticide residue analysis, in terms of selectivity, specificity, and speed of analysis. The extremely high sensitivity of the APGC™ Xevo™ TQ-XS System was demonstrated with reliable detection for almost all analytes at concentrations as low as 0.0003 mg/kg, with an injection volume of 1µL. The method was successfully validated in rice-based baby food using the SANTE guidelines document. The results from analysis of the spikes at both concentrations showed that 91 % and 98 % of the analytes were within the required tolerances for recovery and repeatability, respectively. The method is considered sensitive, specific, accurate, and suitable for the determination of residues of a wide range of GC-amenable pesticides for checking compliance with the specific MRLs set for food intended for infants and young children and has the potential for determination at much lower concentrations.

P-31

Determination of contaminants in dried hemp using LC and GC coupled to MS/MS

Nathaly Reyes-Garcés,[1] Melinda Urich,[1]

[1]**Restek Corporation**, 110 Benner Circle; Bellefonte, PA 16823, USA; Nathaly.reyes@restek.com

Hemp is a class of Cannabis sativa that contains significantly lower levels of tetrahydrocannabinol (THC) (below 0.3%, on a dry weight basis), and may have higher levels of cannabidiol (CBD). This agricultural commodity is being utilized as a source of fiber, protein, and cannabinoids. The 2018 Farm Bill authorized the production and distribution of hemp under federal law, and this led to a significant increase in the development and commercialization of hemp products. Like cannabis and other crops, dried hemp plant material may contain various contaminants that are harmful to humans. For this reason, robust analytical workflows capable of detecting low concentrations of pesticides and other contaminants in hemp and cannabis plant material are highly desired. This work describes a complete workflow for the analysis of diverse contaminants (i.e. pesticides and mycotoxins) in hemp. Briefly, dried hemp samples were pulverized and extracted with acetonitrile. Afterwards, extracts were cleaned-up by passing them through solid phase extraction (SPE) cartridges to remove major interferences. Instrumental analysis was conducted using liquid chromatography (LC), and gas chromatography (GC) both coupled to tandem MS (MS/MS). For GC analysis, an additional clean-up step using dispersive SPE was carried out. The great majority of target contaminants showed recoveries that ranged from 70 to 110% in hemp samples spiked at 10, 50, 100 and 500 ppb. Satisfactory results in terms of LOQ, linearity, accuracy, and precision were also obtained. Overall, the presented workflow provides a reliable and easy approach to quantify multiple contaminants in hemp samples.

P-32

Analysis of Multiclass Multiresidue Pesticides in Pigmented Fresh Produce Using the Enhanced Matrix Removal (EMR) Passthrough Cleanup

Limian Zhao,

Agilent Technologies Inc., 2850 Centerville Rd., Wilmington, DE, 19808

Natural pigments in fresh fruits and vegetables can be highly abundant, which are easily extracted but difficult to remove selectively during sample preparation. The pigment co-extractives result in multiple matrix effect on the detection instrumentation, including matrix ion suppression on LC/MS/MS, matrix interferences on GC/MS/MS, and accumulated matrix deposition on the detection flow path and MS source. The most common pigment removal strategy using Graphitized Carbon Black (GCB) included products can provide efficient pigment removal, but also cause significant loss of sensitive targets such as planar pesticides.

Agilent Carbon S sorbent is an advanced hybrid carbon material with optimized carbon content and pore structure. Compared to GCB sorbent, Carbon S sorbent provides equivalent or better pigment removal, but significantly improves sensitive targets recoveries. Therefore, Carbon S sorbent achieves a better balance between analyte recovery and matrix pigment removal. This advanced sorbent has thus been used in Enhanced Matrix Removal (EMR) products for the efficient and selective passthrough matrix cleanup. Based on the level and pigments from the pigmented produce matrices, different types of products were developed to deliver the best performance and a significantly simplified workflow.

This study evaluated sample preparation using appropriate EMR cartridge cleanup for high chlorophyll and general pigmented fruit and vegetable matrices. Over 200 pesticides were tested for method quantitation performance evaluation, using both LC/MS/MS and GC/MS/MS. The results demonstrated the improved target recovery and reproducibility, the reduced matrix effect and interferences, and the overall increased pass rate for large panel pesticides quantitation in fresh produce.

P-33

Quadrupole Solutions for Testing Residual Solvents and Pesticides in Food Products

Kirk R. Jensen,[1] A. John Dane,[1] Robert B Cody;[1]

[1]**JEOL USA, Inc.**, 11 Dearborn Rd; Peabody, MA 01960, USA; kjensen@jeol.com;

Chemical treatment of food products has a variety of uses, including pesticides to increase crop yields, and chemical extraction for concentrates. The use of chemicals on food products brings with it the need for regulatory testing. In addition to the possible adverse health effects to humans, there are also environmental concerns, such as soil poisoning and bee colony collapse. Two common classes of chemicals tested for in food products are residual solvents and pesticides. Quadrupole gas chromatography-mass spectrometry (GC-MS) is commonly used to measure residual solvents, and triple-quadrupole GC-MS can be used to measure pesticide content. Each method offers robust, sensitive analysis of target analytes. Here, analytical methods for measuring both of these chemical classes are presented along with example data from their respective techniques. Examples in include residual solvents in cannabis and pesticide content in honey. We observed great linearity (R^2>0.98) for all chemical classes measured in both techniques, as well as low peak area RSD (<15%). We also measured instrument detection limits for compounds with strict action limits, and were able to meet the regulatory requirements for the associated methods. Residual solvents observed in a cannabis sample included two compounds from the California Department of Cannabis Control's Class 1 solvents list, and pesticide testing of honey found cabaril, which is highly toxic to bees.

P-34

Determination of Per and Polyfluoroalkyl Substances in Soils Using Carbon S SPE by LC/MS/MS

Matthew Giardina, PhD

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

Graphitized carbon black (GCB) has been used widely in sample preparation for efficient removal of pigments and other matrix interferences. However, GCB may cause the loss of some analytes. Carbon S is an advanced hybrid carbon material with optimized carbon content and pore structure. Compared to GCB, Carbon S provides equivalent or better pigment removal from sample matrices, while significantly improving recovery for some GCB-selective analytes (such as planar pesticides). As a result, Carbon S sorbent provides a better balance between analyte recovery and matrix removal efficiency than traditional GCB sorbent. The Carbon S sorbent is applied in the same SPE cartridge format with the same bed mass as GCB SPE. The Carbon S SPE cartridges can be used as a replacement for the GCB cartridges for applications where SPE methodology is used. This study investigates the post-extraction matrix cleanup of 59 PFAS from loamy sand, reed sedge peat, and topsoil using the Bond Elut Carbon S 250 mg, 6 mL cartridges followed by LC/MS/MS analysis.

P-36

The Challenges to Develop Multi-Class Certified Reference Materials for Food Safety

HuiChen W. Stavros

LGC Standards, 7290-B Investment Drive, North Charleston, SC 29418, USA; huichen.stavros@lgcgroup.com

To support the wide ranges of chemical residue analyses for food safety, approximately 1100 multi-class compounds (insecticides, herbicides, fungicides, metabolites etc.) were selected to develop the current two Smart Solution kits (5 vials in each kit- v700 PestiMix for LC and v400 PestiMix for GC), part of the Certified Reference Materials portfolio with LGC. The challenges when facing to design these large master solutions include sourcing, selectivity, solubility, and stability. Analytical techniques (GC/MS and/or LC/HRMS) of each solution were established. The potential reactivities of each kit were evaluated periodically to improve stability of these Certified Reference Materials. We are constantly expanding our present compound list and continuing to develop additional smart solution kits for food safety analyses.

P-37

Confident analysis of ultra-trace pesticides residues in baby food using triple quadrupole GC

Lori Dolata,[1] Andy Fornadel,[1] Giulia Riccardino [2] Adam Ladak,[3] Paul Silcock,[3] and Daniel Kutscher[4]

[1]**Thermo Fisher Scientific**, 168 Third Avenue, Waltham, MA USA; loriA.dolata@thermofisher.com; [2]Thermo Fisher Scientific, Milan, Italy; [3]Thermo Fisher Scientific, Hemel Hempstead, UK; Thermo Fisher Scientific, Bremen, Germany.

In the EU, MRLs for most pesticide-commodity combinations are set at the default level of 10 µg/kg, but for specific pesticides prohibited in baby foods LOD MRLs are set between 3–8 µg/kg. It is essential laboratories meet these low regulatory limits to ensure the safety of baby foods. In this study, a new GC-MS/MS system, launched in March 2022, was evaluated for the analysis of more than 200 pesticides in complex multi-compositional baby foods. Samples were extracted using the QuEChERS EN method. The overall method was assessed for linearity, instrument detection limit (IDL), limit of quantitation LOQ) and extended robustness over several weeks. The target compounds satisfied the SANTE criteria at the generic default MRL of 10 µg/kg and more than 95% were compliant at < 1 µg/kg. Recoveries for pesticides spiked at 3 µg/kg, were between 70-120% with calculated precision ≤ 10% RSD. Instrument robustness was evaluated over almost four weeks of continuous operation. Repeated injection of various matrix extracts spiked at 10 µg/kg (total number of matrix injections = 500) in randomized order produced results in compliance with SANTE guidelines. After 500 injections the ionization source was removed cleaned and replaced in the system in under 2 hours. The removable AEI ion source for vent-free ion source removal and maintenance, contributed to reduced instrument downtime and improved productivity. More detailed results will be presented in the poster.

P-38

Multi-residue Analysis of Pesticides Regulated by the Colorado State in Hemp Plant Material

Dan Carnevale, Lukas Vaclavik, Cally Maire, Nicholas Grey, John Schmitz, and Grace Bandong

Eurofins Food and Chemistry Testing, 6304 Ronald Reagan Avenue, Madison, WI 53704, USA; lukasvaclavik@eurofins.co.uk

Pesticides are used to protect hemp crops from pests and diseases, and to improve growth yield. Contamination of plants with pesticides may occur not only due to non-optimal direct application, but also due to uptake of pesticides from soil. Modern pesticides are relatively safe and degrade rapidly; however contaminated soil may contain highly toxic persistent compounds, use of which was banned many years prior. Monitoring of pesticide residues in hemp and related products using multi-residue methods with large analyte scope is crucial to ensure consumer safety and compliance with applicable regulations. In the United States, pesticides in hemp are regulated at the state level with target lists and action limits differing largely between states. The most stringent regulation in hemp with action limits for certain analytes as low as 0.01 mg/kg was issued by Colorado. In this study, a QuEChERS-based procedure using both LC-MS/MS and GC-MS/MS techniques was developed and validated for the analysis of 102 pesticides regulated by the state of Colorado in hemp plant material. The validation experiments evaluated selectivity, accuracy, precision and limits of quantification (LOQ). The determined LOQs were at or below the Colorado action limits with spike recoveries within 70-120% and respective relative standard deviations below 20% for the majority of target analytes.

Working Group Abstracts
Reference Materials

RM-01

Increasing the Scope of the Reference Materials Use in Trace Analysis: Highlights for the Second Edition

Patricia Atkins
Spex, an Antylia Scientific Company, 203 Norcross Rd, Metuchen, NJ 08840; patricia.atkins@antylia.com

During the production of the first edition of the RefMaTra, the working group's goal was to cover the best practices for the use of reference materials for trace analyses. In subsequent discussions after its launch, the group felt that the first edition was heavily weighted to the organic analysis field and did not include practices and definitions pertinent to the inorganics trace analysis field. In our second edition, we have been working to include references, examples, and practices unique to the inorganics reference materials users. In this presentation we will take a look at the new elements that are being created for the second edition.

RM-02

Addition of Inorganic Guidance to the Reference Material Use in Trace Analysis Manual

Kevin Kubachka[1]

[1]**USFDA Forensic Chemistry Center**, 6751 Steger Dr.; Cincinnati, OH 45237, USA; kevin.kubachka@fda.hhs.gov;

The North American Chemical Residue Workshop Working Group previously compiled Edition One of the Reference Material Use in Trace Analysis best practices manual in early 2021. This manual primarily focused organic analysis and soon after its release, it was realized that such a guide would benefit the trace element analysis community. Therefore, the NACRW Working Group solicited volunteer subject matter experts in the field of elemental analysis to modify Edition One of the manual to include guidance related to the use of reference materials for inorganic analysis. This presentation will summarize the working group's activities in this area including additional chapters and other significant changes.

RM-03

Use of Reference Materials for the Authentication of Natural Products, Botanicals, and Other Agricultural Materials

Sidney Sudberg,[1] Adam Kuszak,[2]

[1]**Alkemist Labs**, 12661 Hoover Street, Garden Grove, CA, 92841, USA; sidney@alkemist.com;
[2] Office of Dietary Supplements/National Institutes of Health, Bethesda, MD

A new manual designed to provide information on the preparation and use of various types of reference materials for the analysis of identity, authenticity and adulteration of natural products, botanicals, and other agricultural materials, will be discussed.

The goals for this short presentation will be to provide some guidance for a laboratory to create, set-up, maintain, and consistently increase the level of reference material authenticity, while decreasing the measurement uncertainty of the identification and authentication process, in a Fit-for-Purpose environment.

RM-04

Botanicals & Dietary Supplements Collaboration Opportunities

Joe Konschnik, RESTEK Corporation, Bellefonte, PA, USA

Botanicals and Dietary Supplements has been an emerging topic of importance to multiple organizations as the growth of these products soared during the recent pandemic. In additon to NACRW's Reference Materials Working Group which is focused on drafting guidance for Botanicals and Dietary Supplements reference materials, organizations such as AOAC International, a consensus standards development organization and ACIL an independent laboratory trade association have also been discussing and offering additional guidance on testing these products for consumer safety. A brief regulatory overview and an update on activities within these other organizations related to this topic will be presented. Opportunities to collaborate with other organizations to ensure harmonization occurs will also be discussed.

RM-05

Open Forum: Question and Answer Session for NACRW Reference Materials Working Group

Jo Marie Cook[1] and Patricia Atkins[2],

[1]formerly the Florida Department of Agriculture & Consumer Services., 301 Circle Dr., Venice FL 34285, jomarie.cook@outlook.com; [2]Antylia Scientific

The 2022 meeting of the Reference Materials Working Group will be concluded with a question-and-answer forum to discuss the challenges of using reference materials. Topics will include the content of the *1st Edition of the Reference Materials Use in Trace Analysis* manual; the upcoming 2nd Edition which has been updated to include recommendations for use of reference materials in elemental analysis; and plans for *Volume Two: Reference Material Use for Authentication*. Please join us for an informed discussion of the use of reference materials for qualitative analyses including for authentication of foods, botanicals and dietary supplements as well as detection of fraud or adulteration. The input from attendees will help inform authors of the topics of most interest to the NACRW community.

Working Group Abstracts
Veterinary Drugs

VD-01

Progress of the VDR Collaborative Study

Jo Marie Cook[1], Anton Kaufmann[2], Maïwenn Le Floch[3], Steven Lehotay[4], Alejandra Rodriguez[5], Sherri Turnipseed[6], Eric Verdon[3], Jian Wang[7], Jon Wong[8];

[1]NACRW; FL, USA; [2]Official Food Control Authority;Zurich, Switzerland; [3]**Anses-Fougères, National and EU Reference Laboratory for VDR in Food**; Fougères, France; maiwenn.lefloch@anses.fr; [4]USDA Agricultural Research Service; Wyndmoor, USA; [5]University of the Republic; Montevideo, Uruguay; [6]FDA, Animal Drugs Research Center; Denver, CO, USA; [7]CFIA; Calgary, AB, Canada; [8]FDA Center for Food Safety and Applied Nutrition; College Park, MD, USA;

Thanks to the evolution of instruments that increase in sensitivity and detection capacity, it is becoming interesting to implement multi-class methods of several hundred substances in control laboratories. Developments of these kind of methods are already a topic of concern worldwide, and it is relevant to highlight their use oriented towards official control. It is with this in mind that the NACRW Vet Drug Residue Working Group started to talk about the organization in 2019 of an inter-laboratory collaborative study to evaluate the screening practices for veterinary drug residues carried out using various new generation mass spectrometry devices. The main goal of this study is to evaluate and establish general identification criteria for LC-MS methods using empirical data to minimize rates of false positives and negatives. Three "Rounds" are planned by three different leading labs, each of them selecting two different commodities. In each Round, the study coordinator has to prepare and ship a tray of autosampler vials consisting of spiked final extracts and calibration standards. The receiving laboratories have to analyze the extracts in a prescribed sequence with their own method(s) that should include the 30 targeted drug analytes using LC-MS/MS and/or HRMS instrumentation. The organization of the Round 1 started in the fall of 2021 led by the laboratory of Anses-Fougères in France. During this meeting will be presented the evolution of the study over the past twelve months.

VD-02

Stability of Veterinary Drugs

Steven J. Lehotay
U.S. Department of Agriculture, Agricultural Research Service; Wyndmoor, PA 19038; USA. email: steven.lehotay@usda.gov

A stability study was conducted for 27 of the 30 veterinary drugs targeted in the collaborative study being conducted by the Veterinary Drugs Working Group. The drug analytes were monitored over the course of a month at 10, 100, and 1,000 ng/g sample equivalents in the diluted aqueous final extracts for Round 2 in the study (chicken egg and 1/1 bovine liver/kidney). Storage temperatures were -80, -18, 8, and 20 degrees C in the dark. Nearly all of the drugs were stable for at least 30 days in frozen conditions, and few of the analytes were stable at room temperature, as will be shown in the brief presentation.

VD-03

Insights into the new EU Implementing Regulation for Analytical Methods Performance for Veterinary Drug Residue Official Control in Food: CIR (EU) 2021/808

Eric Verdon

Anses-Fougères, National and EU Reference Laboratory for VDR in Food; Fougères, France; eric.verdon@anses.fr

In June 2021 was enforced into the EU a new Commission Implementing Regulation dedicated to update and repeal the old Decisions (EC) 2002/657 and (EC) 98/179 as regard to the performance of analytical methods for residues of pharmacologically active substances used in food-producing animals and on the interpretation of results as well as on the methods to be used for sampling. This presentation will aim at giving some insights on the new approaches inserted in this document and at comparing criteria with those from the 20-year-old former regulation.

VD-04

Open Forum: Question and Answer Session for NACRW Veterinary Drugs Working Group

Jo Marie Cook[1], Anton Kaufmann[2], Maïwenn Le Floch[3], Steven Lehotay[4], Alejandra Rodriguez[5], Sherri Turnipseed[6], Eric Verdon[3], Jian Wang[7], Jon Wong[8];

[1]NACRW, FL, USA; [2]Official Food Control Authority, Zurich, Switzerland; [3]Anses Fougères; 10B Rue Claude Bourgelat, 35133 Javené, France; maiwenn.lefloch@anses.fr; [4]USDA Agricultural Research Service; Wyndmoor, USA; [5]University of the Republic, Montevideo, Uruguay; [6]**FDA, Animal Drugs Research Center**, Denver, CO, USA; [7]CFIA; Calgary, AB, Canada; [8]FDA Center for Food Safety and Applied Nutrition, College Park, MD, USA;

A part of the 2022 Annual Meeting of the NACRW Veterinary Drugs Working Group there will be an open forum intended to facilitate discussion among the audience and core members of the Veterinary Working Group. Questions relating to the ongoing collaborative study and other issues of interest to the group will be included. All interested attendees are welcome to participate in the on-line discussion.