NACRW

NORTH AMERICAN
CHEMICAL RESIDUE WORKSHOP

59th Annual Conference

July 23-26, 2023

Marriott Harbor Beach Resort

Fort Lauderdale, Florida

www.nacrw.org

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Future Meeting Dates

2024 July 14-17
Marriott Harbor Beach Resort
Fort Lauderdale

Dear Attendees, Exhibitors, Sponsors and Guests:

Welcome to the 59th North American Chemical Residue Workshop! After a few years of absence, we are thrilled to be back in person. Whether you are a long-time attendee, an international guest, or a first-time participant, we extend a warm greeting to all. We would like to express our sincere gratitude to our Exhibitors and Sponsors for their generous support, which has made outstanding presentations possible while keeping registration fees affordable for attendees. NACRW has become a favorite event for many, thanks to its fantastic technical sessions, interactive poster presentations, and relaxed atmosphere.

We invite you to join us on Sunday, July 23, for the NACRW Reference Materials and Veterinary Drugs Working Groups. Each Working Group Session will feature interesting presentations and an open forum for discussions and questions.

Our Program Committee has put together a fantastic technical program for this year's workshop. It covers a wide range of chemical residue related subjects and special interest topics, including pesticide testing methods (multi-residue and single residue), updates on sample preparation and processing methodologies for residue testing, cannabis residue testing updates (new methods, challenges, accreditation, and validation topics), emerging environmental and food contaminants like PFAS, and method development and validations. Additionally, we have the Troubleshooting Forum where attendees can ask questions to our panel of experts.

In addition to the oral sessions, we encourage you to attend the poster sessions, visit exhibitors, and attend vendor seminars. The poster authors will be available at designated times to present their posters and engage in discussions. This is a wonderful opportunity to interact with the authors and ask questions. We are pleased to offer student poster awards, sponsored by FLAG Works, Inc. and Restek Corporation. Students will be attending the workshop and will be available at their posters during designated times for conversations and Q&A. We also encourage attendees to visit our exhibitors throughout the workshop to learn more about the products and services they offer for all your testing needs.

I would like to express my deepest appreciation to the fantastic volunteers who have made this event a reality. To this year’s NACRW Organizing Committee, Program Committee, especially Ken Kise, Susie Genualdi, Jo Marie-Cook, and Executive Director Teri Besse, it has been a pleasure working with you, and I am truly grateful for your time and dedication to the workshop. I also want to thank NACRW for providing this opportunity; it has been an incredibly rewarding experience.

We invite you to make the most of your time at NACRW and enjoy all that sunny Florida has to offer. With its warm climate and beautiful beaches, it's the perfect backdrop to enhance your conference experience. Take some time to unwind, soak up the sun, and engage in stimulating discussions about residue testing. We hope you have a fantastic time at NACRW and make lasting memories both inside and outside the conference venue.

Sincerely,

Oscar Cabrices
2023 NACRW Organizing Committee President
Susan Genualdi and Ken Kise
2023 NACRW Program Committee Co-Chairs

The George and Wilma Fong Founders Award

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

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2011 George and Wilma Fong-Founders
2012 Gail Parker
2013 Pat Beckett
2014 Sherry Garris
2015 Jack Cochran
2016 Amy Brown
2017 Jo Marie Cook
2018 Julie Kowalski
2019 Steven Lehotay

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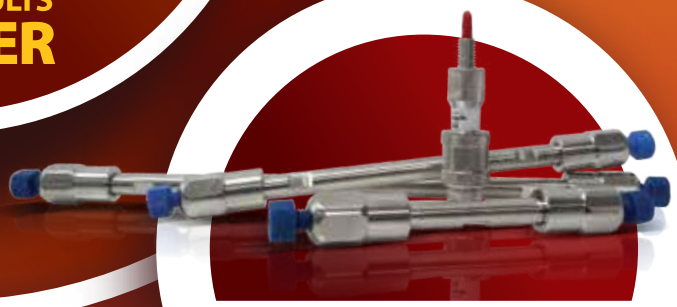

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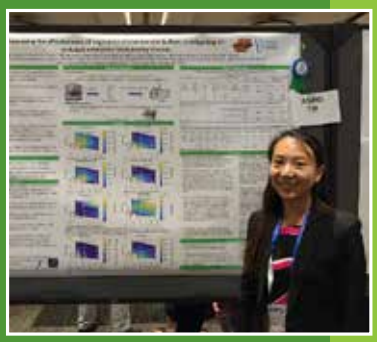

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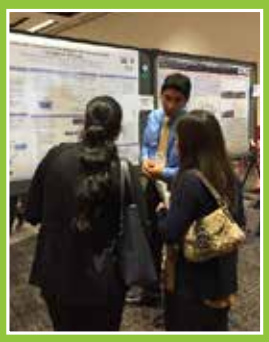

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GENERAL INFORMATION

Registration

Check in once at the registration desk at your earliest opportunity
Sunday - noon – 6:00 pm
Monday - 7:30 am – 5:00 pm
Tuesday - 8:00 am – 4:00 pm
Wednesday - 7:45 am – Noon

KEY to Presentation Numbering System

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

Poster Sessions (Exhibit Hall, Grand Ballroom E-K)

Hang posters Sunday afternoon from 3:00 pm to 6:00 pm or Monday morning from 7:00 am to 9:30 am.
Take down posters between 12 noon to 2:00 pm on Wednesday

Posters may be viewed any time Exhibition is open

Poster Session A (odd#) authors must be at their posters from 11:00 am – noon on Monday and 3:10 – 3:55 pm on Tuesday
Poster Session B (even#) authors must be at their posters from 3:10 pm – 3:40 pm on Monday and 11:00 – noon on Tuesday

Poster Prizes

Two poster prizes of $200 each will be awarded this year, and the same poster/author(s) are eligible to win both prizes. The People's Choice Poster Award will be determined by popular vote of attendees, and the Judges Choice Poster Award will be determined by the poster committee. The criteria used in each case will be importance of the study, quality of the science, and its presentation (including oral discussion and abstract). **Attendees must place their votes in the ballot box by noon on Wednesday.** ***Be sure to write your name on the back of your voting ticket for the chance to win a door prize.***

Exhibition (Grand Ballroom E-K)

Sunday: A welcome reception with light hors d'oeuvres and an open beer and wine bar will be held from 6:30 - 8:00 pm. If you would like to try the **"Strawberry OrbiRITA"** cocktail stop by the ***Thermo Fisher Scientific*** booth to get a ticket for a free drink. **Booth #28**
Monday: 10 am - 1 pm and 2:30 pm - 4 pm
Tuesday: 10 am - 1 pm and 2:30 pm - 4 pm
Wednesday: 10 am - 12 noon

Coffee and Breaks

Coffee will be available each morning in the Caribbean Ballroom foyer (please refer to the times in the conference program). There will also be mid-morning and afternoon refreshment breaks each day. The Monday and Tuesday mid-morning and afternoon breaks, as well as the Wednesday mid-morning break, will be served in the Exhibition Hall (Grand Ballroom E-K). On Wednesday afternoon, the break will be served in the Caribbean Ballroom foyer.

Announcements

Moderators will make general announcements from the podium. If you need to have an announcement made, please go to the conference registration desk or see the onsite audio-visual team in the meeting room, and give them your annoucement.

Mobile App

Thanks to the Platinum Sponsors we have a mobile app to view the program, see who is attending the meeting, and connect with them via the app. You should have received an email just prior to the conference with a link to download the app to your mobile phone along with a link that will automatically log you in.

Door Prizes

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

Get to Know Your Sponsor

Participate in the "Get to Know Your Sponsor" quiz and win an Apple iPad tablet. A quiz will be provided to you in your registration bag. Simply take the quiz to each sponsor booth, get the right answer and the sponsor will place a sticker on your quiz. After you have completed the quiz, return it to the registration desk no later than Wednesday, July 26, at 1:30 pm. We will be announcing the winner Wednesday afternoon.

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

Oral and Poster presenters are encouraged to contribute original research and/or review articles to the Journal of Agricultural and Food Chemistry for a special section related to the 2023 NACRW. Please inform **Teri Besse (teri@nacrw.org), by September 1, 2023,** if you intend to submit an article. Authors will then be invited by JAFC to submit their manuscripts electronically online through the JAFC website with a deadline of November 30, 2023.

Copies of Presentations

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

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

Meeting Website

www.NACRW.org - the website includes information on current and future NACRW meetings, as well as archives going back a few years.

Meeting Evaluations

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

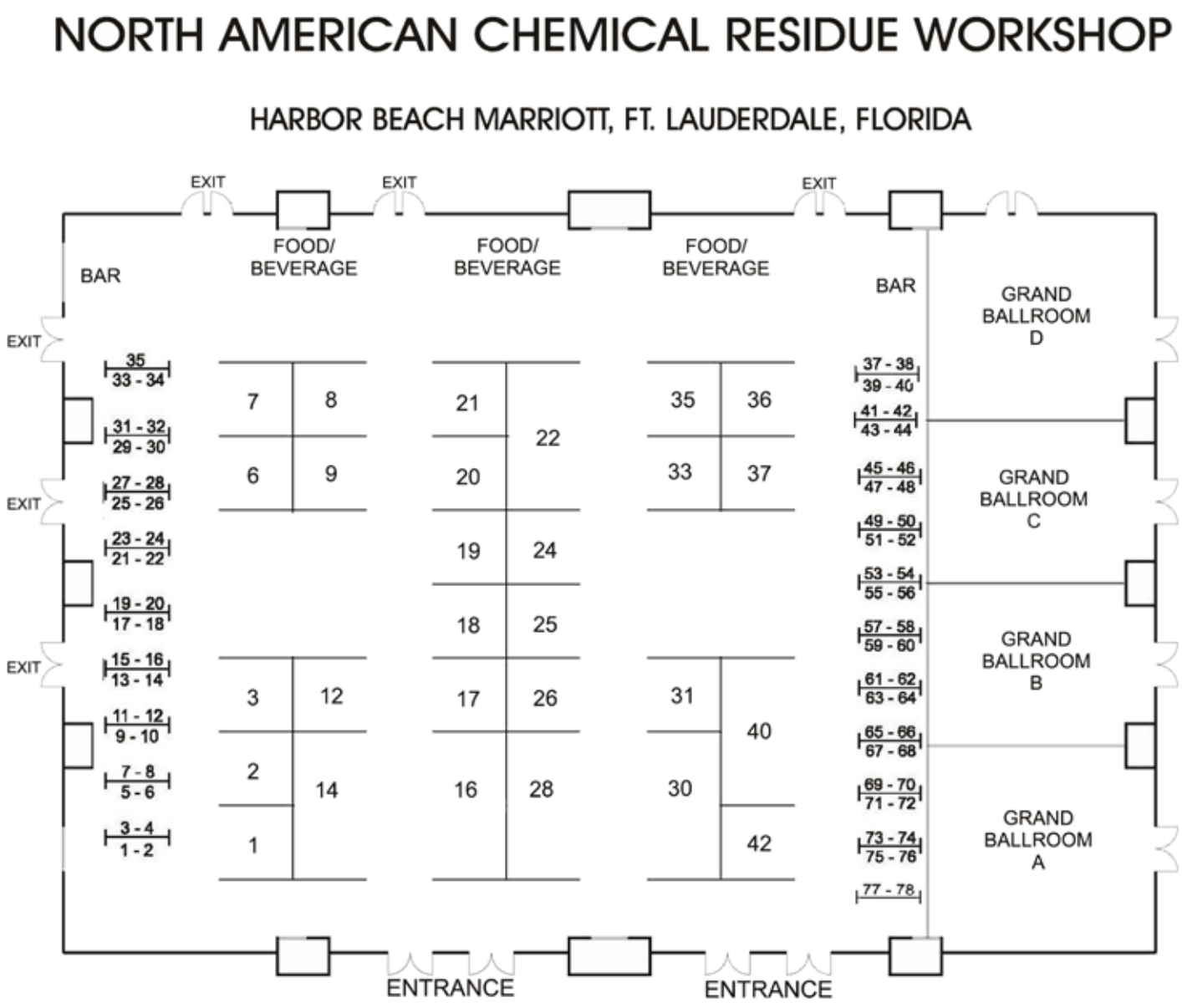
A BIG THANK YOU TO ALL OF OUR VOLUNTEERS, SPONSORS & EXHIBITORS!
The workshop would not be possible without your valuable assistance.

MARK YOUR CALENDAR FOR THE 2024 NACRW

July 14-17 Fort Lauderdale Marriott Harbor Beach Resort & Spa

EXHIBITION HALL
AND
POSTER SESSIONS

Location: Grand Ballroom E-K, 3rd level



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Cannabis Science & Technology, LCGC and Spectroscopy
Booth # 40

AccuStandard, Inc.
Booth # 42

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William Fatigante
Applications Scientist

Join Us for Breakfast!

Wednesday, July 26th in Grand Salons A-D, Level 3, 7:15-8:15am

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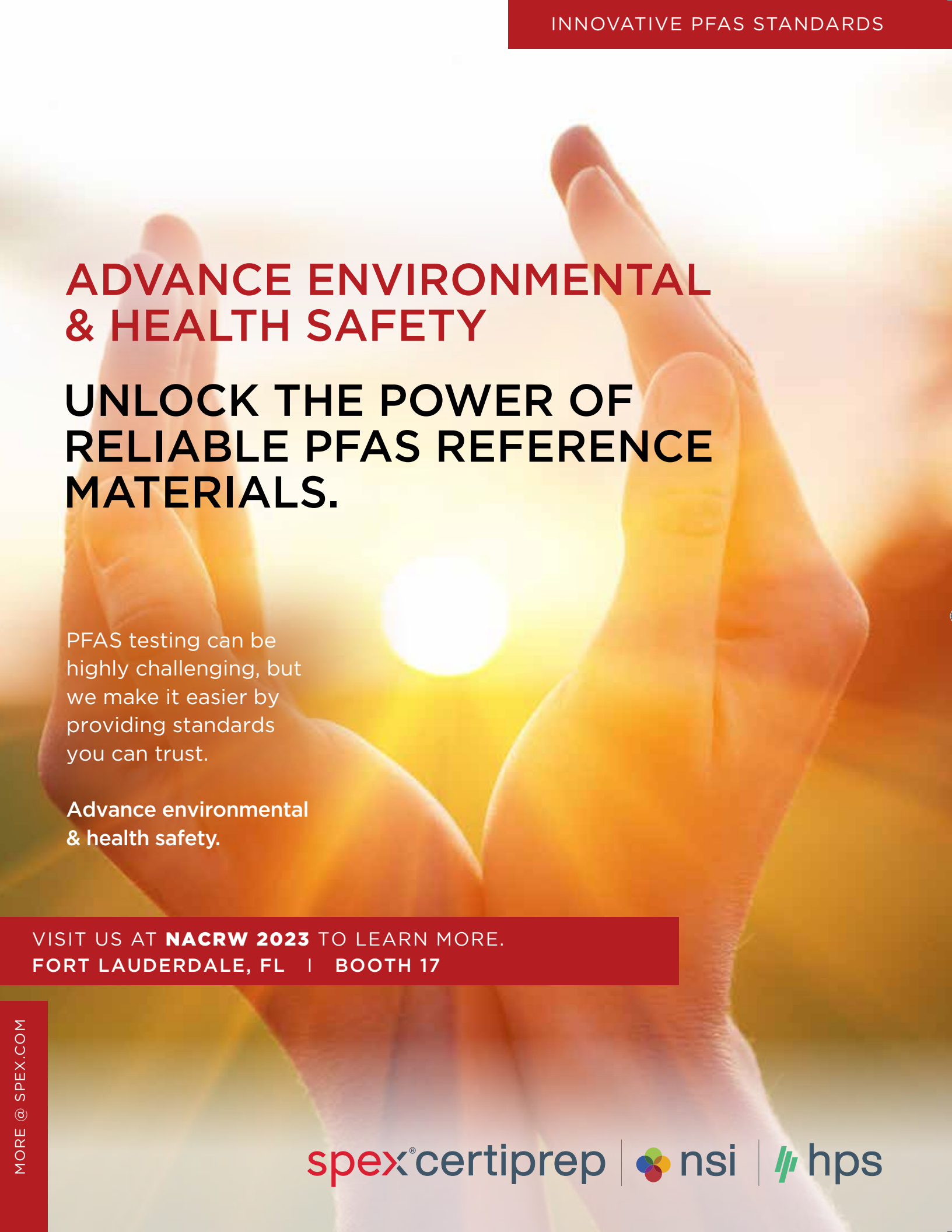

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(PRE-REGISTRATION IS REQUIRED)

Please sign up at the meeting registration desk

V-1 Monday, July 24, 2023 7:15-8:15 am WATERS CORP.

Location: Grand Ballroom A-D, 2nd floor

Pesticides, Natural Toxins, and PFAS – Hot Topic Updates for Trace Contaminant Workflows

Emily Rose Britton, PhD, Principal Marketing Manager for Food & Natural Products, ***Waters Corporation***
Gordon Fujimoto, PhD, Chemical Analysis Group Leader, Americas MS Application Lab, ***Waters Corporation***

Testing labs play an important role in the supply chain – they are essential in keeping the food supply safe, and yet consumers are often unaware of their existence! News coverage and rising concerns about contaminated food and water supplies have pulled back the curtain and shed light on the critical value of testing labs. Testing demand is on the rise, and new regulations and safety threats pose an opportunity for laboratories. So, what's your contaminant of choice? Pesticides, natural toxins, PFAS, all of the above? Join the Waters vendor seminar to hear about workflow and technology innovations for trace contaminant testing and a recent interlaboratory study and performance evaluation for the detection of PFAS in fish using LC-MS/MS.

V-2 Monday, July 24, 2023 12:15-1:15 pm SCIEX

Location: Grand Ballroom A-D, 2nd floor

From PFAS to ergot alkaloids: residue testing working beyond pesticides

Analysis of ergot akaloids in a variety of simple and complex matrices by liquid chromatography-tandem quadrupole mass spectrometry

Julie Brunkhorst[1], Emilee Easter[1], Kristen Moseley[1], Kendra Adams[2]
[1] ***Trilogy Analytical Laboratory*** *[2] SCIEX*

Ergot alkaloids are mycotoxins produced by fungi most commonly associated with rye, wheat, barley and oats. On January 1st of 2022, the EU published maximum levels of ergot sclerotia and ergot alkaloids at the new maximum of of 0.2-0.5 g/kg. To support import/export compliance a method was developed to quantitate ergot alkaloids in rye, wheat, barley, oats, finished feeds, feed ingredients and other matrices. The method developed utilizes liquid chromatography coupled with tandem quadrupole mass spectrometry and includes ergometrine, ergosine, ergotamine, ergocornine, ergocryptine, ergocristine, and ergovaline, as well as their corresponding -inine isomers.

continued on next page

From the tap to our home: the analysis of perfluoroalkyl and polyfluoroalkyl substances (PFAS) across their lifecycle

Holly Lee[1], Jianru Stahl-Zeng[1], Karl Oetjen[1], and Craig Butt[1]
*[1] **SCIEX***

Recent detections of perfluoroalkyl and polyfluoroalkyl substances (PFAS) in apparel, cosmetics and food contact materials have elevated PFAS from an environmental issue into a public health concern given the potential toxicological impact of long-term exposure. As such, the fate of PFAS from use to disposal must be holistically considered by investigating these chemicals across their entire life cycle. We will present workflows for the quantitation and identification of PFAS in food, firefighting foams and cosmetics – products that cross much of the PFAS life cycle. This includes the use of electron activated dissociation (EAD) from the ZenoTOF 7600 system as an orthogonal fragmentation mechanism for structural elucidation.

V-3 Tuesday, July 25, 2023 7:15-8:15 am THERMO FISHER SCIENTIFIC

Location: Grand Ballroom A-D, 2nd floor

NEW ANALYTICAL TOOLS FOR PESTICIDE RESIDUES IN FOOD CONTROL

*Prof. Amadeo Rodriguez Fernandez-Alba, **EURL-FV**, EU Reference Laboratory for pesticide residues in Fruits and Vegetables, **University of Almería**, Spain*
*Richard Fussell, Market Development Manager, **Thermo Fisher Scientific**, Hemel Hempstead, UK*

Amadeo will discuss the practical implementation of recent instrument advancements to address the golden triangle of pesticide residue analysis: (i) Sensitivity, (ii) Scope and (iii) Speed. For example, the improvement in sensitivity provided by high-end triple quads to mitigate matrix effects and new high-resolution MS systems which enable simultaneous and sensitive acquisition in both targeted and non-targeted analysis in a very consistent way. The sensitivity achieved is still less than that of triple quad MS, but they still achieve default values of 10 ppb. Dual-column chromatography with two columns connected to one MS not only improves utilization of the MS time but can also reduce the chromatographic analysis time to increase sample throughput without compromising selectivity. Alternatively, it can facilitate optimization of eluent compositions for both positive and negative detection modes, simultaneously, to improve LOQs for an increased number of pesticides without increasing the analysis time.

Richard will close with a brief update on developments in the analysis of polar cationic pesticides, options to either reduce or to eliminate the usage of helium as a GC carrier gas, and a new single software with some unique features for efficient data processing of pesticides data acquired by LC-MS, GC-MS and IC-MS.

V-4 Tuesday, July 25, 2023 12:15-1:15 pm AGILENT TECHNOLOGIES

Location: Grand Ballroom A-D, 2nd floor

Hydrogen for Pesticide Residue Analysis: How to Convert GC/MS/MS Analysis from Helium to Hydrogen and Meet the MRLs

Lorna De Leoz, PhD, Global Food Segment Manager, ***Agilent Technologies***

This presentation will address the key strategies for pesticide analysis using GC/MS/MS with hydrogen as the carrier gas that allow for maintaining sensitivity to meet MRLs. The optimized setup with hydrogen showed improved chromatographic resolution and allowed for precisely matching the retention time with helium. The HydroInert and HES sources were shown to provide best sensitivity and preserve spectral fidelity even for the compounds highly prone to reacting with hydrogen in the source by minimizing or preventing such undesirable reactions. As a result, the same MRM transitions, with the same collision energies for the targets eluting at the same retention times as with helium could be used with hydrogen carrier gas, streamlining the transition from helium to hydrogen. The presented method allowed for quantitation of over 90% of target pesticides at or below the default MRL of 10 ppb in spinach.

V-5 Wednesday, July 26, 2023 7:15-8:15 am BRUKER

Location: Grand Ballroom A-D, 2nd floor

Higher Throughput, Higher Accuracy, and Higher Sensitivity – New Mass Spectrometric Solutions for Food Analysis

William Fatigante, Applications Scientist, ***Bruker Applied Mass Spectrometry***

Bruker Applied Mass Spectrometry continues to develop new solutions to overcome ever-increasing challenges of food safety and quality analysis. Through persistent innovation and diligent refinement of its technology, Bruker has achieved some remarkable improvements in the most critical aspects of analytical workflows: speed, accuracy, and sensitivity. From highly efficient VIP-HESI ion source to extremally fast DART JumpShot ionization; from very robust EVOQ triple quadrupole mass spectrometers to state-of-the-art timsTOF systems; from highly automated software for routine analysis and intelligent software to complete out-of-the-box TargetScreener 4D solution – Bruker technology addresses challenges of the day and futureproofs laboratories for the challenges of tomorrow.

Join us for breakfast and this short overview presentation to learn about these and other Bruker innovations for food safety and quality analysis.

V-6 Wednesday, July 26, 2023, 12 noon-1:00 pm RESTEK

Location: Grand Ballroom A-D, 2nd floor

Analysis of ultrashort-chain and short-chain PFAS in potable and non-potable waters

Shun-Hsin Liang, Senior Scientist II, ***Restek Corporation***

While the current standard tests are focusing on the PFAS with carbon chains of C4 and up using reversed-phase liquid chromatography (RPLC) methodologies, more studies have shown the prevalence and high levels of ultrashort-chain (USC) PFAS (C1-C3) in environmental aquatic systems. Therefore, a simple and reliable LC method is necessary to fulfill the need for standard analytical workflows of USC PFAS. This presentation will discuss a proposed ASTM method development (WK80687) for C1 to C4 carboxylic acid and sulfonic acid PFAS analysis in tap waters, bottle waters, and wastewaters.

Large Volume Injection of Pesticides in Matrix Using Low Pressure Gas Chromatography

Jana Hepner, Senior Scientist, ***Restek Corporation***

Concurrent Solvent Recondensation Large Sample Volume splitless injection (CRS-LV, or LVI) is a sample technique that overcomes the limitation of the maximum sample volume to 1 – 2 µL valid for classical splitless injection. The LPGC configuration with the restrictor/guard column lends itself to the requirements of the CRS-LV and has a potential to improve the sensitivity of the detection. Large volume injection of matrix extract (acetonitrile and acetonitrile – toluene) samples were evaluated in range of 1 – 25 µL for peak shapes and the relationship between the peak area and injection volume was established. The calibration parameters were compared for 5 µL, 15 µL and 25 µL injection.

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Oral and Poster Presenters

(alphabetical order by Last Name)

First Name	Last Name	Presentation
Rakan	Alajmi	P-18
Christine	Allen	P-27
George	Antonious	P-16
Kevin	Armbrust	O-31
Cristy	Barringer	P-65
Laura	Basirico	O-32
Bjorn	Berendsen	O-15
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MEETING PROGRAM

Sunday, July 23, 2023

8:00 am-4:00 pm — **Short Course:** — **Key West/Palm Beach**
"Squeezing more out of Laboratory Analyses with QA/QC"
Dr. Jens Andersen, Department of Chemical and Forensic Sciences, Botswana International University of Science and Technology, Palapye, Botswana

12 noon-6:00 pm — **Registration** — **Caribbean Foyer**

1:00-5:00 pm — **Exhibitor set-up** — **Grand Ballroom E-K**
3:00 - 6:00 pm — **Poster Board set-up** — **Grand Ballroom**

1:00-2:30 pm — **NACRW Reference Materials Working Group** — **Caribbean Ballroom 1-3**
Developing Guidelines for Use of Reference Materials
Co-chairs: Patricia Atkins and Jo Marie Cook

1:00-1:10 pm — **Jo Marie Cook**, Florida Department of Agriculture, retired, Venice, FL, USA
Introduction to the Activities of the RMWG

1:10-1:25 pm — **Patricia Atkins**, SPEX, Metuchen, NJ, USA
Updates in Edition Two of the *Reference Material Use in Trace Analysis* Guide.

1:25-1:45 pm — **Reference Materials for the Study of Natural Material Identification, Authenticity and Adulteration**

- **Adam Kuszak**, National Institutes of Health, Office of Dietary Supplements, MD, USA
  Objectives of the Authenticity Working Group
- **Sidney Sudberg,** Alkemist Labs, CA, USA
  Case Study of the Development and Use of a Botanical Reference Material

1:45-2:30 pm — **Jo Marie Cook -Moderator**, Florida Department of Agriculture, FL, USA, retired
Open Forum

2:45-4:15 pm — **NACRW Veterinary Drug Residue (VDR) Working Group** — **Caribbean Ballroom 1-3**
VDR Collaborative Studies
Co-chairs: Eric Verdon and Sherri Turnipseed

2:45-3:05 pm — **Anton Kaufmann**, Official Food Control Authority Of The Canton Of Zürich, Zürich, Switzerland
Comparison of the Low verus High Resolution-based Confirmation Criteria for Veterinary Chemical Residues in Food Control

3:05-3:25 pm — **Maiwenn Le Floch** and **Eric Verdon**,
ANSES, the French AGENCY for Food, Environmental and Occupational Health & Safety Laboratory of Fougeres, France
Progress of the Veterinary Drug Residue Collaborative Study, 2022-2023 (Round 1)

3:25-3:45 pm — **Development of HRMS Food Residue Megamethods**

- **Steven Lehotay**, USDA ARS, Wyndmoor, PA, USA
  Extraction
- **Jian Wang,** Canadian Food Inspection Agency, Calgary, Alberta, Canada
  Jon Wong, USFDA, Center for Food Safety and Applied Nutrition, College Park, MD, USA
  Pesticides
- **Sherri Turnipseed,** USFDA Animal Drugs Research Center, Denver, CO; USA
  Veterinary Drugs

3:45-4:05 pm	**Jo Marie Cook -Moderator**, Florida Department of Agriculture, retired, Venice, FL, USA **Open Forum**	
4:05-4:15 pm	**Eric Verdon and Sherry Turnipseed** **Summary and Closing Remarks**	
4:30-6:00 pm	**NACRW Working Groups General Session** **Future Projects** **Co-chairs: Patricia Atkins and Jo Marie Cook**	**Caribbean Ballroom 1-3**
4:30-4:40 pm	**Jo Marie Cook**, Florida Department of Agriculture, retired, Venice, FL, USA **Introduction to the Working Groups and their Terms of Reference.**	
4:40-4:50 pm	**Patricia Atkins, SPEX** **Goals of the Authenticity Working Group and Beyond**	
4:50-5:00 pm	**Simon Hird**, Waters Corporation, Wimlsow, Cheshire, United Kingdom **Challenges and Solutions to Troubleshooting**	
5:00-5:10 pm	**Alex Chao**, U.S. EPA, Durham, NC, USA **Reference Materials for Non-Target Analyses**	
5:10-6:00 pm	**Simon Hird**, Waters Corporation, Wimlsow, Cheshire, United Kingdom **Open Forum**	
6:30-8:00 pm	**Welcome Reception, Exhibit Hall**	**Grand Ballroom**

Monday, July 24, 2023

7:00-9:30 am	Poster Board Set-up	**Grand Ballroom E-K**
7:15-8:15 am	**Waters Corp. Vendor Seminar** *(pre-registration required)*	**Grand Ballroom A-D**
7:30 am-5:00 pm	**Registration**	**Caribbean Foyer**
7:45-8:15 am	**Early Morning Coffee**	**Caribbean Foyer**
8:15-8:40 am	**Opening Remarks**	**Caribbean Ballroom**
8:15-8:30 am	**Sherry Garris, Chair, FLAG Works, Inc. Board of Directors**	
8:30-8:35 am	**2023 NACRW President, Oscar Cabrices**	
8:35-8:40 am	**2023 NACRW Program Co-Chairs, Susie Genualdi** and **Ken Kise**	
8:40-10:45 am	**SESSION 1:** ***sponsored by*** **Pesticide Testing Methods (Multi Residue and Single Residue)** **Co-Chairs: Rodney Bennett and Kelsey Powers**	ThermoFisher SCIENTIFIC
8:40-8:45 am	**Session Sponsorship - Thermo Fisher Scientific** **Ed George**, Food And Beverage Applications Manager	
8:45-9:10 am **O-01**	**Simon Hird**, Waters Corporation, Wimlsow, Cheshire, United Kingdom **The Advantages of Using Atmospheric Pressure Ionization (APGC technology) for Determination of Pesticide Residues in Baby Foods using GC-MS/MS**	

9:10-9:35 am
O-02
Jian Wang, Canadian Food Inspection Agency, Calgary, Alberta, Canada
Practical Applications of nDATA for Quantitation and Identification of Mostly Incurred Pesticides and Screening of Less Frequently found Residues in Fruits and Vegetables using UHPLC/ESI Q-Orbitrap Data Independent Acquisition

9:35-10:00 am
O-03
Wiley Hall, USDA-ARS, Parlier, CA, USA
Considerations and Progress in Developing Multiresidue Analysis Methods for the Residues of Postharvest and Preplant Fumigants.

10:00-10:25 am
O-04
Lorna De Leoz, Agilent Technologies, Wilmington, DE, USA
Hydrogen as a Sustainable Alternative to Helium in Pesticide Residual GC/MS/MS Analysis: Method Translation, Optimization, and Application to Analyzing Pesticides in Pigmented Foods

10:25 – 10:50 am
O-05
Kazuaki Iijima, The Institute Of Environmental Toxicology, Joso, Ibaraki, Japan
Relationship Between Surface Properties of Fruits and Fruiting Vegetables and Residue Levels of Pesticides by Foliage Application

10:50 am - noon
Exhibition and Poster Opening — **Grand Ballroom E-K**

11:00 am - noon
Poster Session A (authors present for odd #s) — **Grand Ballroom E-K**

12 noon
Lunch on your own

12:15 - 1:15 pm
SCIEX Vendor Seminar *(pre-registration required)* — **Grand Ballroom A-D**

1:25 - 3:10 pm
SESSION 2: *sponsored by* Agilent — **Caribbean Ballroom**
Updates on Sample Preparation and Processing Methodologies for Residue Testing
Co-Chairs: Jana Hepner and Raegyn Taylor

1:25-1:30 pm
Session Sponsorship - Agilent Technologies
M. Lorna De Leoz, Ph.D., Global Food Segment Manager

1:30 – 1:55 pm
O-06
Michael Ebitson, ThermoFisher Scientific, Bedford, NH, USA
Improved Automated Sample Preparation for Persistent Organic Pollutants using Parallel Gas Assisted Accelerated Solvent Extraction and Automated Solvent Concentration

1:55 – 2:20 pm
O-07
James (Mike) Farrow, USFDA, Atlanta, GA, USA
FDA's Laboratory Flexible Funding Model — Three Years and Counting

2:20 – 2:45 pm
O-08
Jerry Mueller, NOW Foods, Bloomingdale, IL, USA
Streamlined Sample Preparation for LC-MS/MS and GC-MS/MS Multi-residue Pesticides Analysis in Botanicals and Essential Oils Using Novel Mixed Mode Cartridge Passthrough Cleanup

2:45 – 3:10 pm
O-09
Steven Lehotay, USDA ARS, Wyndmoor, PA, USA
Evaluation of a New Cryogenic Device for Comminution of 1 kg Bulk Food Samples for Analysis of Contaminants in <1 g Test Portions

3:10-3:40 pm
BREAK (Exhibition & Posters) — **Grand Ballroom E-K**
Poster Session B (authors present for even #s)

3:40-5:10 pm	**SESSION 3:** *sponsored by* RESTEK **Cannabis Residue Testing Updates** **Co-Chairs: Geoff DuBrow and Alexandria Bush**	**Caribbean Ballroom**
3:40-3:45 pm	**Session Sponsorship – RESTEK**	
3:45 – 4:05 pm **O-10**	**Matthew Noestheden**, SCIEX, Salmon Arm, British Columbia, Canada **Suspect Screening of Cannabis for 288 Mycotoxins and their Metabolites using High-Resolution Mass Spectrometry**	
4:05 – 4:25 pm **O-11**	**Ronald Francisco**, Florida Department of Agriculture and Consumer Services, Tallahassee, FL, USA **Residual Solvents Testing in Florida in CBD and Hemp Oil Products by HS-GC-MS**	
4:25 – 4:45 pm **O-12**	**Geoffrey Dubrow**, U.S. Food and Drug Administration, College Park, MD, USA **An Improved, Validated LC-UV/FLD and LC-MS/MS Method for the Quantification of Cannabinoids in Hemp-Derived Ingestible Products**	
4:45 -5:10 pm **O-13**	***Keynote speaker*** – **Peter Morton**, Texas A&M University, College Station, TX, USA **Proceed with Caution: Addressing the Opportunities and Hazards of *Sargassum* Inundation**	
6:30-9:30 pm	**SOCIAL EVENT Dinner – Marriott Harbor Beach** **One-hour reception provided by NACRW; Games after dinner on the Ocean Terrace**	**Oceanside Ballroom & Terrace**

Tuesday, July 25, 2023

7:15-8:15 am	**Thermo Fisher Scientific Vendor Seminar** *(pre-registration required)*	**Grand Ballroom A-D**
8:00 am - 4:00 pm	**Registration**	**Caribbean Foyer**
8:00 am – 8:30 am	**Early Morning Coffee**	**Caribbean Foyer**
8:40 am - 10:45 am	**SESSION 4:** *sponsored by* Waters™ **Emerging Environmental and Food Contaminants** **Co-Chairs: Matt Noestheden and Lukas Vaclavik**	**Caribbean Ballroom**
8:40 – 8:45 am	**Session Sponsorship – Waters Corp.** **Emily Rose Britton, Ph.D.**, Principal Marketing Manager - Food & Natural Products Americas Field Marketing	
8:45 – 9:10 am **O-14**	**Wendy Young**, USFDA, College Park, MD, USA **Updates and Expansion of FDA's Analytical Method for PFAS Detection in Food and Feed**	
9:10 – 9:35 am **O-15**	**Bjorn Berendsen**, Wageningen Food Safety Research, Wageningen, Netherlands **PFAS Analysis in Food Products - Challenges, Methods and Results**	
9:35 – 10:00 am **O-16**	**Yelena Sapozhnikova**, USDA, Wyndmoor, PA, USA **Analysis of PFAS in Globally Sourced Consumer Food Packaging by Targeted and Non-Targeted Approaches**	

10:00 – 10:25 am **O-17**	**Anton Kaufmann**, Official Food Control Authority Of The Canton Of Zürich, Zürich, Switzerland **Fast and Easy Nontargeted Detection of Pfas in Complex Food Matrices**	
10:25 – 10:50 am **O-18**	**Charles Neslund**, Eurofins Lancaster Laboratories Environment Testing, Lancaster, PA, USA **The Analysis for PFAS in Food and Feed: An Evaluation using the FDA Quechers Application**	
10:55 am - noon	**Exhibition and Posters**	**Grand Ballroom**
11:00 am - noon	**Poster Session B** ***(authors present for even #s)***	**Grand Ballroom**
12 noon	**Lunch on your own**	
12:15-1:15 pm	**Agilent Vendor Seminar** *(pre-registration required)*	**Grand Ballroom A-D**
1:30-3:10 pm	**SESSION 5:** **Method Development and Validations** **Co-Chairs: Katie Carlos and Yelena Sapozhnikova**	**Caribbean Ballroom**
1:30 – 1:55 pm **O-19**	**Katerina Mastovska**, AOAC INTERNATIONAL, Rockvile, Maryland, USA **Method Development and Validation – AOAC's Perspective and Initiatives Focused on Residues and Contaminants**	
1:55 – 2:20 pm **O-20**	**Alex Chao**, U.S. EPA, Durham, NC, USA **Validation of Non-Targeted Analysis Methods: Establishing and Communicating Confidence in Results**	
2:20 – 2:45 pm **O-21**	**Lawrence Zintek**, US EPA R5, Chicago, Illinois, USA **Interlaboratory Validation of ASTM D8421, Standard Test Method forDetermination of Per- and Polyfluoroalkyl Substances(PFAS) in Aqueous Matrices by Co-solvation followed by Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS)**	
2:45 – 3:10 pm **O-22**	**Jens Enevold Thaulov Andersen**, Botswana International University Of Science And Technology, Palapye, Botswana, Botswana **Meta Analysis of Pesticide Residues in Food Products Shows that the Associated Quality Assurance may need Improvements**	
3:10-3:55 pm	**BREAK (Exhibition & Posters)** **Poster Session A (authors present for odd #s)**	**Grand Ballroom**
4:00 - 5:00 pm	**SESSION 6:** **Laboratory Troubleshooting Forum** **(The classic MS debates, sample preparation challenges, analysis challenges)** **Moderators: Scott Krepich and Simon Hird**	**Caribbean Ballroom**
5:05-6:00 pm	**NACRW Organizing Committee Meeting** **open to all attendees**	**Caribbean Ballroom**
6:30-10:30 pm	**Shuttle service to Las Olas Blvd.** ***sponsored by VisitLauderdale Convention & Visitors Bureau***	**Outside -Entrance at Caribbean Ballroom**

Wednesday, July 26, 2023

7:15-8:15 am	**Bruker Vendor Seminar** *(pre-registration required)*	**Grand Ballroom A-D**
7:45 am-noon	**Registration**	**Caribbean Foyer**
7:45-8:15 am	**Early Morning Coffee**	**Caribbean Foyer**
8:25-10:00 am	**SESSION 7:** *sponsored by* SCIEX **Advanced Analytical Techniques for Residue Testing** **Co-Chairs: Jian Wang and Wiley Hall**	**Caribbean Ballroom**
8:25-8:30 am	**Session Sponsorship – SCIEX** **Karl Oetjen, Ph.D.**, SCIEX	
8:30 – 8:50 am **O-23**	**Julien Parinet**, Anses, Montreuil, France **Liquid chromatographic retention time prediction models to secure and improve the feature annotation process in high-resolution mass spectrometry**	
8:50 – 9:10 am **O-24**	**Jon Wong**, US FDA, College Park, MD, USA **Multilaboratory Collaborative Study of a non-Target Data Acquisition for Target Analysis (nDATA) Workflow for the Screening of >1000 Pesticides in Fruits and Vegetables**	
9:10 – 9:30 am **O-25**	**Erik Rangel Rivera**, University of California at Davis, Davis, CA, USA **Capillary electrophoresis-, normal atmospheric pressure-, and particle beam - mass spectrometry for residue applications**	
9:30 – 9:50 am **O-26**	**Amadeo Fernández-Alba**, EURL-FV University of Almería, La Cañada de San Urbano, Almería, Spain **Cutting Edge Methods for Pesticide Residue Analysis**	
10:00-10:45 am	**Student Scholarship Award Presentations**	**Caribbean Ballroom**
10:45 - noon	**BREAK (Exhibition & Posters)**	**Grand Ballroom**
12 noon	**Lunch on your own**	
12:00 - 1:00 pm	**Restek Vendor Seminar** *(pre-registration required)*	**Grand Ballroom A-D**
1:05 - 2:45 pm	**SESSION 8:** **Natural Toxins Residue Testing** **Co-Chairs: Wade Huang and Kai Zhang**	**Caribbean Ballroom**
1:05 – 1:30 pm **O-27**	**Dojin Ryu**, University of Idaho, Moscow, ID, USA **Significance of Ochratoxin A in Agricultural Commodities and Processed Food Products**	
1:30 – 1:55 pm **O-28**	**Weili Xiong**, FDA CFSAN, College Park, MD, USA **Analysis of Ergot Alkaloids in Cereal-based Food Products using LC-MS/MS**	
1:55 – 2:20 pm **O-29**	**Wade (Ishuo) Huang**, US FDA, College Park, MD, USA **Development of an LC-MS/MS Method for the Detection of Microcystins in Marine and Estuarine Shellfish**	

2:20 – 2:45 pm O-30	**Pearse McCarron**, National Research Council Canada, Halifax, Nova Scotia, Canada **Development of a Cyanobacterial Dietary Supplement Reference Material for Multiple Classes of Cyanotoxins**	
2:45-3:15 pm	**BREAK**	**Caribbean Foyer**
3:15-4:55 pm	**SESSION 9:** **General Topics** **Co-Chairs: Jens Andersen and Sareeta Nerkar**	**Caribbean Ballroom**
3:15 – 3:40 pm O-31	**Kevin Armbrust**, LSU -Dept Of Environmental Sciences, Baton Rouge, LA, USA **Harmonizing pesticide tolerances through regulatory capacity building: The CERSA-USDA FAS collaboration**	
3:40 – 4:05 pm O-32	**Laura Basirico**, Louisiana State University, Baton Rouge, LA, USA **Novel Low Sulfur Distillate and Residual Fuel Oils as Emerging Environmental Contaminants**	
4:05 – 4:30 pm O-33	**Richard Jack**, Phenomenex, San Jose, CA, USA **Method Performance using dual WAX/GCB and GCB/WAX SPE Formats for Draft EPA Method 1633**	
4:30 – 4:55 pm O-34	**Natalia Soares Quinete**, Florida International University, North Miami, FL, USA **Beyond the traditional targeted analysis approach: Comprehensive screening of PFAS in South Florida environments by Non-Targeted Analysis**	
4:55 - 5:10 pm	**Poster Awards and Closing** Award Presentations by André De Kok and Brittany Holmes, Poster Co-Chairs	**Caribbean Ballroom**

Thursday, July 27, 2023

7:30 am – 12:30 pm	**Concurrent User Meetings**	
	Agilent (7:30 – 9:30)	**Caribbean 4**
	Thermo Fisher Scientific (7:30 – 9:30)	**Caribbean 6-7**
	Waters (10:30 am – 12:30 pm)	**Caribbean 2-3**

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Monday, July 24 at 12:15 p.m. | featuring Trilogy Analytical Laboratory

ORAL PRESENTATION ABSTRACTS

O-01

The advantages of using atmospheric pressure ionization (APGC technology) for determination of pesticide residues in baby foods using GC-MS/MS

Simon Hird,[1] Janitha De-Alwis,[1] David Gould,[1] Stuart Adams,[1] and Frank Dorman;[2]

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

We developed a method based on gas chromatography-tandem mass spectrometry (GC–MS/MS) for the determination of over 200 pesticides in baby food. Extracts of cottage pie baby food were prepared using a modification of the QuEChERS CEN Method 156624, whereby the dispersive solid-phase extraction (dSPE) step was replaced with pass through SPE for removal of fats, phospholipids, and pigments. The extremely high sensitivity of GC-MS/MS utilizing atmospheric pressure ionization source (APGC™ technology) enabled the reliable detection for almost all the analytes at concentrations as low as 0.00025 mg/kg (0.25 µg/kg), even when the injection volume was 1µL. The method was successfully validated using the SANTE guidelines. The results from analysis of spikes at 0.0005 and 0.001 mg/kg showed 87 and 93% of the analytes were within the required tolerance for recovery and 97 and 99.5% for repeatability. The method is considered suitable for the determination of residues of a wide range of GC-amenable pesticides in baby food, for checking compliance with the specific MRLs set for food intended for infants and young children in Europe and by the food industry for due diligence and brand protection.
Volatility and interruptions to supplies of helium have led to increased costs and shortages, which has prompted evaluation of other gases as alternative carrier gases for GC-MS/MS. Nitrogen, which is cheaper and readily available, can be easily used as the GC carrier gas with the APGC source. Comparable performance was obtained with nitrogen by using a smaller diameter column and scaling the parameters accordingly.

O-02

Practical Applications of nDATA for quantitation and identification of mostly incurred pesticides and screening of less frequently found residues in fruits and vegetables using UHPLC/ESI Q-Orbitrap data independent acquisition

Jian Wang[1], Daniel Leung[1], Willis Chow[1]

[1] **Canadian Food Inspection Agency**, Calgary Laboratory, 3650-36th Street N.W., Calgary, Alberta, T2L 2L1, Canada; jian.wang@inspection.gc.ca

This study presents a cost-effective approach that uses UHPLC/ESI Q-Orbitrap non-target data acquisition for target analysis (nDATA) to improve the monitoring program efficiency. The application allows for checking compliance by quantitating the most incurred pesticide residues while maintaining a wide testing scope by simultaneously qualitative screening for less frequently found pesticides. The scope of the method for quantitation and screening was determined according to historical monitoring data. We examined CFIA monitoring data collected between 2014 to 2019, from 11415 fruit and vegetable samples tested by both GC-MS (295 pesticides) and LC/MS/MS (187 pesticides). This study then validated the nDATA method for quantitation of 126 incurred pesticides, which had been found at least once in the selected time frame, in terms of overall recovery, intermediate precision and measurement uncertainty. The study confirmed the effectiveness of using nDATA along with Compound Database to screen a total of 374 pesticides, which included 126 commonly found pesticides as well as an additional 248 pesticides that had not been detected in six years, and the validation was based on 95% detection rate. The nDATA method was able to determine 77.6% (374 pesticides) of the 482 pesticides in fruits and vegetables in one single 14-min data acquisition using UHPLC Q-Orbitrap Full MS/vDIA. The remaining ~100 pesticides are GC-amenable which have to be analyzed by GC-MS. Using nDATA for both quantitation and screening in a single analysis, there is no need to prepare the full calibration curves for all 374 pesticides but only the incurred ones. The nDATA approach will reduce the cost on pesticide standards, and save resources to maintain standards and to review and process data.

O-03

Considerations and Progress in Developing Multiresidue Analysis Methods for the Residues of Postharvest and Preplant Fumigants.

Wiley A. Hall, 4th[1]

[1]**USDA-ARS-SJVASC**, 9611 S. Riverbend Ave; Parlier, CA 93648, USA; wiley.hall@usda.gov

Retail / customer demands for produce free of pesticide residues continues to fuel a push for exporters to perform more testing for a greater number of compounds. This pressure, in turn, drives a need to reduce the costs of pesticide testing by combining single residue methods into multiresidue methods whenever possible. Recent work related to an ongoing effort to improve analytical methodology for the detection of fumigants ranging from gasses, like methyl bromide (MB), sulfuryl fluoride (SF) or hydrogen cyanide, to volatile compounds such as ethylene oxide (EtO), propylene oxide (PPO), methyl isothiocyanate (MITC) and 1,3-D, all in a single analysis will be presented. Headspace solid-phase microextraction (HS-SPME) analysis will be shown to accurately quantify multiple fumigant residues in foodstuffs without lengthy sample preparation or the use of solvents. Critical considerations for this method, including sample preparation, preparation of matrix matched standards, column selection, and analytical challenges such as prevention of analyte loss through degradation or evaporation, will all be addressed. The potential to use analytical techniques such as capillary electrophoresis (CE) or ion chromatography (IC) to quantify the inorganic degradants of fumigant compounds will also be discussed.

O-04

Hydrogen as a Sustainable Alternative to Helium in Pesticide Residual GC/MS/MS Analysis: Method Translation, Optimization, and Application to Analyzing Pesticides in Pigmented Foods

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

The choice of carrier gas is critical in GC/MS/MS analysis because it directly affects method performance, such as sensitivity, chromatographic resolution, ability to use databases, and speed. Helium has been the carrier gas of choice for GC/MS analysis.
Hydrogen is a sustainable alternative to helium, which can provide superior chromatographic performance at lower cost. However, the undesirable chemical reactions between target analytes and hydrogen occurring in the GC/MS flow path and especially in the MS EI source created obstacles to the migration to hydrogen.
This presentation discusses the successful conversion of the GC/MS/MS pesticide residue analysis from helium to hydrogen carrier gas. GC method translation technique and retention time locking allowed for observing the same retention times with hydrogen as with helium streamlining the transition. The optimized GC method parameters coupled with effective sample preparation enabled sensitivity sufficient to meet pesticide maximum residue limits (MRLs).
Most importantly, appropriate EI source design, was determined for use with hydrogen carrier gas. Three EI source configurations were evaluated, two of which minimized the undesirable in-source reaction, hence, preserving spectral fidelity and allowing for using the MRM transitions developed with helium carrier gas.
In summary, the presented method uses the same retention times and MRM transitions as the original helium method. The method performance was evaluated using 200 pesticides in a pigmented spinach matrix in terms of sensitivity, calibration performance, and method ruggedness. The method was able to detect pesticide residues at levels below the MRLs, offered excellent chromatographic resolution and good calibration performance.

O-05

Relationship between surface properties of fruits and fruiting vegetables and residue levels of pesticides by foliage application

Kazuaki Iijima,[1] Shinobu Hikino,[1] Kei Kondo,[1] Tomonari Yajima,[1] Yoshiki Wakasone[1] and Takahiro Watanabe[2]

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With the development of food transportation systems such as refrigeration technology, there are a wide variety of fruits and fruiting vegetables on our dining table, year by year. However, with regard to unfamiliar new varieties and/or imported fruits, which are called minor crops, there is the problem of little residue data to evaluate their influence for human health, in comparison with conventional major crops. As mentioned above, we conducted simple experiments such as dipping experiments and statistical analysis of residue data from crop field trials by converting them into residue amounts per fruit surface area (μg/cm^2) instead of conventional concentration basis (mg/kg). The purpose of this study is to clarify the difference in residue levels between minor crops and major crops with different surface properties (ex. Hairly- vs. Hairless-kiwifruit, Muskmelon with rough-ridged vs. Watermelon with flat-smooth surface). The comparative results based on experimental values in this presentation contain valuable scientific knowledge that is necessary for a deeper understanding of residual data. Part of this work was supported by a Health Sciences Research Grant from the Ministry of Health, Labor, and Welfare of Japan.

O-06

Improved Automated Sample Preparation for Persistent Organic Pollutants using Parallel Gas Assisted Accelerated Solvent Extraction and Automated Solvent Concentration

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Accelerated solvent extraction is a high-temperature and high-pressure extraction technique that is widely used in environmental, chemical and food analysis. New advancements have been made in accelerated solvent extraction which allows for a more efficient and controlled extraction to allow for better extraction over traditional methods and devices. Gas assisted extraction is a new method employed for extracting analytes of interest from a solid or semi-solid matrix, that uses a mixture of a liquid solvent at a constant flow rate. Gas assisted extraction can be more effective as compared to static extraction methods. In addition, when done in parallel, this new technique yields many advantages. Furthermore, the solvent evaporation process is fully incorporated into the automated process.
In this presentation, we discuss a new parallel gas assisted extraction for pursuing extractions and by quantitation for PFAS and persistence organic pollutants (POPs). We present here performance data of the new method for analyte extraction and evaporation in the same platform from soil samples for different POP. Unlike traditional methods such as liquid-solvent extraction followed by N2 stream evaporation, the fully automated solvent extraction and evaporation system saves time, solvent, and labor, while ensuring high reproducibility and productivity for analytical testing.

O-07

FDA's Laboratory Flexible Funding Model — Three Years and Counting

Mike Farrow and Lauren Yeung, US FDA, Office of Human and Animal Food Laboratory Operations, External Laboratories

The presentation will review the first three years of the FDA's Laboratory Flexible Funding Model (LFFM), including overall structure, specific tracks (projects), and accomplishments. Under LFFM, state partners conduct surveillance of human and animal foods for microbiological and chemical hazards. Impacts and outcomes of surveillance for regulatory actions and signals evaluation will be described. LFFM provides structured support for state laboratory capacity and capability development. This presentation will provide examples of implementing harmonized methods and supporting method development and validation efforts, and how these enhanced capabilities/capacities are leveraged by state and federal partners. Finally, the presentation will share the life cycle of an LFFM year, including planning, pivoting, metrics tracking. In sum, LFFM activities provide mutual benefit to federal and state stakeholders and a tangible example of an operationalized national integrated food safety system.

O-08

Streamlined Sample Preparation for LC-MS/MS and GC-MS/MS Multi-residue Pesticides Analysis in Botanicals and Essential Oils Using Novel Mixed Mode Cartridge Passthrough Cleanup

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A big challenge in routine multi-residue pesticides analysis in botanical extracts is the significant matrix complexity and variety and difficulty to clean. The abundant matrix interferences cause the significant matrix effect on LC/MS/MS and contribute a lot of matrix interferences on GC/MS/MS. Typically, a graphitized carbon black (GCB)/NH_2 cartridge was applied for sample crude extract cleanup, to remove pigments and acids co-extractives. However, the GCB caused the planar pesticides loss, and necessitated an elution solvent containing toluene to recover the planar analytes. This resulted in the separate preparations for LC-MS/MS and GC-MS/MS, which are costly, time consuming, and high risks for human errors or contamination. Therefore, there is a need for a versatile sample preparation, which can adequately cleanup the matrix without irreversibly trapping analytes of interest.
A novel mixed mode cartridge passthrough cleanup was developed utilizing EMR cartridges after sample extraction. It demonstrated as a simplified workflow and provided the comprehensive matrix cleanup, including pigments, acids, oily and other hydrophobic interferences, without trapping planar and other sensitive analytes. The new method enabled using one sample preparation for both LC-MS/MS and GC-MS/MS analysis, saving analyst's time and lab cost. This method improved recoveries and repeatability for more analytes, and thus resulted in higher acceptance rate for pesticides determination in challenge matrices such as ginger root powder, peppermint essential oil, and other botanical extracts. The streamlined procedure allows for a flexible and robust workflow to handle a wide range of botanicals and produce consistent data to support routine multi-residue pesticides analysis.

O-09

Evaluation of a new cryogenic device for comminution of 1 kg food samples for analysis of contaminants in <1 g test portions

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Like a chain is only as strong as its weakest link, the overall accuracy (and speed) in the analysis of chemical contaminants in foods is limited by the least accurate (and slowest) step in the protocol. In practice, sample processing (comminution) of bulk samples to generate the test portion for analysis is nearly always the analytical performance (and throughput) limiting step. Regulatory entities worldwide call for comminution of 1 kg bulk samples for most food commodities in pesticide residue monitoring, which requires large food processors. Several studies have demonstrated that cryogenic comminution with liquid nitrogen or dry ice leads to best performance, but until now, no devices had yet been commercialized to accommodate such large watery samples. A newly introduced "cryo-blade" device has a 5 L bowl to contain about 30 mL chunks of pre-cut 1 kg samples at room temperature, which are processed using liquid nitrogen automatically controlled from a coupled Dewar, or 1 kg of dry ice can be used instead. Higher sample preparation efficiency is attained by taking smaller test portions for analysis, but accuracy is decreased when test portions become too small. In this study, the cryo-blade was evaluated for QuEChERSER sample preparation and both GC-MS/MS and LC-MS/MS analysis of many spiked and incurred pesticides in 1 kg samples of watermelon, grapes, and green beans processed using liquid nitrogen, dry ice, or at room temperature. Initial results show acceptable consistency of results for test portions as low as 0.5 g, and full results will be presented.

O-10

Suspect screening of Cannabis for 288 mycotoxins and their metabolites using high-resolution mass spectrometry

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Mycotoxins are toxic secondary metabolites produced by fungi that can cause acute and chronic illness in humans and animals. More than 300 mycotoxins are known, but the most common that threaten human health include aflatoxin (AF), deoxynivalenol (DON), ochratoxin A (OTA), fumonisin (FB), zearalenone (ZEN) and T-2 toxin (T2), all of which are widely found in agriculture crops, such as grains, oils and their products.
Cannabis is an agricultural crop destined for human consumption both medicinally and recreationally in a variety of formats. In the United States, Cannabis is still federally classified as a "Schedule 1 Drug," leaving states to decide if and which mycotoxins to monitor in Cannabis products. Given the lack of consistent regulation, typically only five mycotoxins are tested for in Cannabis products, Ochratoxin A, Aflatoxin B1, B2, G1, and G2, even though Cannabis is no less subject to mycotoxin contamination than other crops. In this study, 100 samples that failed microbial testing, but were negative for the five mycotoxins that are typically the focus in Cannabis testing, were analyzed using high-resolution mass spectrometry and screened against a spectral library consisting of 288 mycotoxins and metabolites. Results from this study show that additional screening for mycotoxins is likely needed to ensure consumer health.

O-11

Residual Solvents Testing in Florida in CBD and Hemp Oil Products by HS-GC-MS

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The acceptance of cannabidiol (CBD) products is very prevalent across the United States. Though the efficacy and legality

of these psychoactive compounds such as tetrahydrocannabinol (THC) and its isomers, are still a question for concern, it does not seem like an industry that is slowing down. More manufacturers are increasing production and infusing THC and CBD in a varying number of matrices. These products come in many forms such as vape oils, lotions, gummies, chocolates, and many others. Some of these products are easily available in gas stations and grocery stores. To infuse these therapeutic compounds into a multitude of products, a solvent extraction is performed on the cannabis flower. Many solvents that are used for this extraction are toxic if consumed via inhalation or via ingestion. Since these are the most common ways of transmission, regulation of the number of solvents as well as other hazards in CBD products is monitored by the Florida Department of Agriculture and Consumer Services (FDACS). FDACS is looking into residual solvents in CBD products by headspace gas chromatography via mass spectrometry (HS-GC-MS). The lab has been able to identify and quantify many residual solvents in CBD and hemp oil products well below their established tolerance levels with great accuracy and precision. From multiple routine sample batches, it was observed that correlation values for solvents of interest were >0.996 and average aggregate spike recoveries of 106%, and average RT drift is as low as 0.423% RPD.

O-12

An Improved, Validated LC-UV/FLD and LC-MS/MS Method for the Quantification of Cannabinoids in Hemp-Derived Ingestible Products

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Over a short period of time, the United States has seen a rapid increase in interest and availability of cannabidiol (CBD) products and other products derived from hemp. Sampling and testing of marketed hemp-derived products is an important component of FDA's work because little is known about the amounts of CBD and other cannabinoids in these products. However, due to the diversity of products, range of matrix interferences, and widely varying cannabinoid concentrations, including minor cannabinoid residues, accurate quantification of cannabinoid content is challenging. To address this challenge, a method was developed pairing QuEChERS with UHPLC-UV/FLD and optional MS/MS analysis. The reported method provides accurate, sensitive, and rapid quantification of 17 cannabinoids, including Δ8-THC, Δ9-THC, Δ10-THC, and CBD, in a wide array of matrices. Sample preparation workflows were harmonized for different product categories, including gummies, edibles, and oils, allowing for a simplified sample preparation workflow. The use of fluorescence detection at pH 6.5 provided enhanced sensitivity and selectivity for both neutral and acidic cannabinoids compared to UV absorption. For quantification of cannabinoids at sub-part-per-million levels and confirmation of UV/FLD quantifications, a paired MS/MS analysis was developed. A single laboratory validation of the method was performed. This method provides a valuable tool for researchers and testing laboratories analyzing hemp-derived products.

O-13

Proceed with Caution: Addressing the Opportunities and Hazards of *Sargassum* Inundation

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The Great Atlantic *Sargassum* Belt has been steadily increasing in size since 2011, inundating coastlines throughout the Gulf of Mexico and Caribbean with mounds of decaying macroalgae for federal, state, and coastal communities to address. Current explanations for this recent rapid expansion implicate an increasing supply of unbalanced nutrients

to the ocean from multiple sources. This nutrient supply has driven dramatic changes in the *Sargassum* biomass composition over the last 40 years: an increase of the N composition of *Sargassum* by 35% and a concurrent decrease of P by 44%. The causes are global in scale and beyond the resources and responsibilities of a single state or nation to solve. Because *Sargassum* provides a critical habitat for numerous marine species in the open ocean, the approaching *Sargassum* inundation cannot be addressed until it reaches the shore. Removing the material from the beach costs governments millions of dollars annually while disrupting coastal ecology (e.g., seagrass beds and sea turtle nesting grounds) and human economies (e.g., tourism and fisheries). Leaving *Sargassum* to decay naturally could release potentially toxic concentrations of hydrogen sulfide, ammonia, and arsenic, posing additional risks to human health and the environment.
On the other hand, creative entrepreneurs view the *Sargassum* problem as an opportunity. Economically profitable proposals include converting the nutrient-rich biomass into fertilizer or animal feed. However, many of the same hazards remain, especially the potentially toxic concentrations of arsenic. These chemical hazards should be thoroughly considered before economic exploitation rushes to "make lemonade from lemons."

O-14

Updates and Expansion of FDA's Analytical Method for PFAS Detection in Food and Feed

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To continually assess human exposure to per- and polyfluoroalkyl substances (PFAS) from food and feed, the FDA has extended its' analytical method to include additional PFAS and expanded the method to additional matrices. The method was first extended to include four additional long-chain perfluorocarboxylic acids (PFCAs), which are known to be present in seafood. A seafood collection of 81 highly consumed seafood products were analyzed for 20 PFAS using the updated analytical method. A wide range of concentrations were observed among the seafood samples. These ranged from below the method detection limit to the highest concentration of 23 µg kg^{-1} for the sum of PFAS in one of the canned clam samples. The analytical method has also been extended to silage and animal feed matrices and used in studies in collaboration with the USDA for the estimation of Holstein cow exposures from water and feed. More recently, the method has been expanded to 30 analytes that have the potential to uptake in food and to further align with other US and international agencies. An investigation into equivalent solid phase extraction (SPE) cartridges and their suitability for PFAS analysis (with 30 analytes) has also been performed. Four different cartridges have been investigated with multiple matrices using the FDA single lab method validation protocol.

O-15

PFAS analysis in food products – challenges, methods and results

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The European Food Safety Authority has recently published a new opinion (i.e. risk assessment) in which they lowered the tolerable weekly intake for the perfluoroalkyl substances with a special focus on PFOA, PFOS, PFNA and PFHxS. This has a severe impact on the required performance of analytical methodologies to detect, confirm the identity and quantify these compounds in edible matrices. For example, currently for PFOA in milk a level of quantification (LOQ) in the low pg/g is required for effective exposure analysis, which is approximately a factor of 100 lower than before. Besides the lowering of the required LOQ of the analytical methods, the scope of PFASs that are relevant is expanding. PFOS and PFOA are currently still the PFAS occurring the most in Dutch waters and food products, but that might change in the future. For instance, as a result of the ban of PFOA by the United Nations alternative PFASs are of interest. In The

Netherlands, the use of heptafluoropropylene oxide dimer acid (HFPO-DA), also called GenX, is used as an alternative and the trimer acid (HFPO-TA) is suspect of occurring. But, besides these, thousands of potentially harmful PFAS might be relevant as the landscape seems to be continuously changing. Therefore, new approaches such as non-target screening, total fluor analysis and total oxidizable precursor assay (TOPA) are being implemented aiming to detect 'unknown' PFAS. The current challenges in analytical approaches for PFAS analysis will be discussed. These include the clean-up of food products and the concentration of extracts to achieve the low detection limits, chemical background that occurs especially for PFOA and short chain PFAS, and finally severe in source fragmentation that occurs for some alternative PFAS and compromises detectability. Data of some surveys will be presented as well as examples of the approaches that are used for detecting unknown PFAS.

O-16

Analysis of PFAS in globally sourced consumer food packaging by targeted and non-targeted approaches

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The purpose of this study was to investigate both occurrence and levels of PFAS in food packaging products, such as greaseproof papers, paperboard trays, wrappers, and cardboard, that represent food choices for different consumer groups. Globally sourced food packaging samples represented food contact materials (FCMs) from the US and 23 other countries. Both targeted analysis (before and after a total oxidizable precursor (TOP) assay) and non-targeted analysis were employed to characterize the extent of PFAS contamination in food packaging extracts. Overall, 84% of food packaging samples had detectable levels of at least one targeted PFAS with 6:2 fluorotelomer phosphate diester (6:2 DiPAP) most frequently detected. Other frequently detected substances were PFHxS, PFHpA and PFDA (found in 15–17% of samples). Shorter chain perfluorinated carboxylic acids PFHpA, PFPeA and PFHxS were at the highest levels in a given sample at 51.3, 24.1 and 18.2 ng/g, respectively. Average ∑PFAS levels increased one order of magnitude after the TOP assay oxidation suggesting the presence of PFAS precursors in tested FCMs. Of the analyzed food packaging extracts, 25 samples with the highest amounts of measured PFAS were selected for subsequent migration experiments with food simulants. PFHxS, PFHpA, PFHxA and 6:2 DiPAP were detected in food simulants in five samples with levels increasing over the ten day migration period. To estimate dietary intake of PFAS from food packaging samples, tolerable weekly intake (TWI) was calculated using the concentration of a PFAS in food simulant and ranged from 0.0006 ng/kg body weight/week for PFHxA exposure in tomato packaging to 1.12 ng/kg body weight/week for PFHxS exposure in cake paper. These values are below the established European Food Safety Authority (EFSA) maximum TWI of 4.4 ng/kg body weight/week for the sum of PFOA, PFNA, PFHxS and PFOS.

O-17

Fast and easy nontargeted detection of PFAS in complex food matrices

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Conventional targeted technologies detect only a fraction of the potentially present PFAS compounds.
Hence there is a high motivation to use non-targeted methodologies. It is the absence of fluorine isotopes which makes the mass spectrometry based nontargeted detection of PFAS difficult. Yet, each hydrogen replaced by a fluorine atom not only increases the mass defect, but also makes the molecule heavier. Therefore, the idea was conceived that not the mass (*m/z*) or the mass defect (md) of a particular ion should be studied, but that these measurement-based values should be divided (normalized) by the number of carbons (C) present in the studied ion. The number of carbons present in an ion can be readily estimated by comparing the ion abundance of the monoisotopic peak versus the first isotopic peak.
Plotting md/C versus *m/z*/C strongly discriminates exogenous PFAS from endogenous compounds. This was

demonstrated by analyzing fish tissues (muscle and liver). The high-resolution mass spectrometry based chromatograms were deconvoluted and the two mentioned parameters were calculated for each of the thousands of extracted chromatographic peaks (features). The nontargeted methodology detected PFAS down to sub µg/kg levels. Detected was a homologue not present in the standard mix, fragments (CO_2 loss) and previously unreported PFAS ms adducts. Unlike other techniques, the simple approach does not rely on fragmentation data, neutral losses, assumption of homologous series or the availability of spectra libraries. The proposed methodology is not only simple to use but has already been integrated into a commercially available ms data processing software and has been independently and comprehensibly evaluated in a recently published peer reviewed paper.

O-18

The Analysis for PFAS in Food and Feed: An Evaluation using the FDA Quechers Application

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The concern for PFAS as a class of emerging contaminants of concern has escalated significantly over the last several years. Much of the effort from EPA is centered on ingestion from drinking water and those factors that impact drinking water such as wastewater discharge and surface water. More recently effort has been spent on the monitoring and analysis of PFAS in biosolids, as the beneficial use of biosolids for agricultural applications has been shown to be a route for contamination of agricultural livestock and products. Additionally, the use of PFAS related products in food contact materials (FCM) has raised concern as one more route for PFAs ingestion. With the many possible routes for ingestion, the question becomes, “are PFAS in food and is the consumption of food a path of ingestion for these environmental contaminants?”
In the absence of a standardized procedure, contract laboratories had made adjustments to their in-house, user defined procedures for the analysis of food. In 2019, FDA first published a procedure for the analysis of PFAS in foods using the Quechers procedure. This presentation will discuss the application of the Quechers protocol for food analysis and compare the effectiveness of the procedure to techniques widely used prior to the application of Quechers to PFAS analysis. We will share results of the application to a variety of foods and make recommendations for the improvement of the application using techniques like isotope dilution. Lastly, we will try to answer the question, “is PFAS in food really a problem?”.

O-19

Method Development and Validation – AOAC’s Perspective and Initiatives Focused on Residues and Contaminants

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AOAC INTERNATIONAL is well-known for its *Official Methods of Analysis*℠ (OMA) program, which develops official methods that are recognized worldwide. In addition to that, AOAC also validates proprietary kit-based testing methods and instrument-based laboratory-developed methods/application notes through *Performance Tested Methods*℠ (PTM) and *Reviewed & Recognized*℠ (R^2) programs, respectively. New OMA initiatives start with the development of *Standard Method Performance Requirements* (SMPRs®), which are voluntary consensus-based standards developed by AOAC’s working groups to define minimum performance characteristics that a given method should meet. SMPRs are used by method developers to develop, optimize and validate methods. They are also used by AOAC’s expert review panels to evaluate methods that are being considered for the official method status. There are several recent AOAC initiatives focused on chemical residues and contaminants, including, for example, projects on PFAS, heavy metals, pyrrolizidine alkaloids, acrylamide, chlorate & perchlorate, glyphosate & metabolites, furan & alkyl furans, or various contaminants in natural color additives or in cannabis and cannabis-derived products. These initiatives are at different stages of the

standard and official method development process, with various opportunities for the analytical community to get involved and contribute to the SMPR development, submit or review methods etc. This presentation will share AOAC's perspective on method development and validation, highlight initiatives of interest to the NACRW audience and discuss ways how people can get actively involved in the AOAC process.

O-20

Validation of Non-Targeted Analysis Methods: Establishing and Communicating Confidence in Results

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Targeted analytical methods have long been the standard for monitoring chemicals that may be of significant exposure potential and/or health risk. Given an increasing need to rapidly identify new and data poor contaminants, monitoring approaches have shifted towards discovery- and screening-based methods like non-targeted analysis (NTA) which have a wider scope of chemical coverage. Validation of targeted methods involves demonstrating confidence in the reported results via specific analyses metrics of reference standards. NTA methods often are run without these accompanying standards; as such, validation of these methods is not only a matter of demonstrating confidence in the results, but defining their scope. This talk will cover different approaches brought together in order to "validate" NTA results, including software tools for processing NTA data, databases and applications that provide chemical identity data in lieu of reference standards, and standardization of NTA practices in order to accurately communicate scope and limitations of NTA results. Demonstration of validation of NTA results will be presented on recent exposure monitoring studies performed. This abstract does not necessarily reflect Agency policy.

O-21

Interlaboratory Validation of ASTM D8421, Standard Test Method for Determination of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Matrices by Co-solvation followed by Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS)

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ASTM conducted an inter-laboratory study (ILS) to establish precision and bias for ASTM D8421 that employs liquid chromatography and tandem mass spectrometry (LC-MS/MS) to measure PFAS compounds. The focus of this ILS was on analyses of PFAS contaminants in various non-potable water matrices. The objectives of this study were to 1) characterize the performance of ASTM Standard D8421 in multiple laboratories 2) evaluate and, if appropriate, revise the quality control (QC) acceptance criteria in the method. The ultimate objective is that EPA propose and promulgate the ASTM Standard in 40 CFR part 136 for use in EPA's Clean Water Act programs. This presentation will elaborate on the ILS results including precision and bias from the study.

O-22

Meta Analysis of Pesticide Residues in Food Products Shows that the Associated Quality Assurance may need Improvements

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Many publications concerned with the analysis of pesticides in various matrices using different advanced methodologies are characterized by listing results with a multitude of molecular species. The number of available pesticides in agricultural products is very extensive, which poses challenges to deliver full method validations for every single method and every single compound. A recent forensic investigation indicates that it is important to pay attention to the performance characteristics of every single species when analyzed with LC-MS/MS. Such investigations remain to be performed for GC-MS/MS and other technologies that are commonly used for these purposes. Hence, this poses a risk to the interpretation of the accrued data of individual species, which are subject to the influence of matrix effects that, alternatively, may also be identified as high levels of uncertainty of measurement that could have been overlooked in during the method validation. Contemporary methods of quality assurance involve the construction of uncertainty budgets but that tends to be an almost insurmountable task for the simultaneous analysis of numerous species with methods of chromatography. Owing to the importance of pesticide analysis in relation to consumer products numerous publications are available with supplementary data that allow for further considerations with respect to the method validation and corresponding uncertainty of measurement. In many instances, were reported very low levels of pesticides in consumer products, which instigated spiking to the products with measurand concentrations that were close to the limit of quantification (LOQ). Such low spiking levels ought to exhibit large uncertainties of measurements.

O-23

Liquid chromatographic retention time prediction models to secure and improve the feature annotation process in high-resolution mass spectrometry

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The discovery of unexpected, emerging contaminants and their by-products and metabolites or newly produced pesticides through suspected and non-targeted approaches is gaining more and more attention and open new horizons in many fields such as food safety, ecotoxicology, environment, health… Nevertheless, the process of moving from annotation to identification can be time-consuming, complex and fraught. The prediction of liquid chromatographic retention times (RT) by different approaches can be an useful and operational way to efficiently discriminate and select between several molecular formulas and between several molecular structures.
The development of quantitative structure-retention relationship (QSRR) models, which are types of models allowing to establish a link between a chemical structure and a property, here the chromatographic retention time, requires an adequate selection of molecular descriptors necessarily obtained based on a chemical structure known. This requires also a selection of the best machine learning/IA algorithm and its optimization.
Here we will present different strategies for the selection of descriptors, different types of machine learning/IA algorithms according to the different situations we are confronted with. These strategies may vary depending on the level of information/annotation we have. We will conclude by proposing a methodology based in part on QSRR to improve and secure the annotation process as it has been published by Schymanski and colleagues in 2014.

O-24

Multilaboratory Collaborative Study of a non-Target Data Acquisition for Target Analysis (nDATA) Workflow for the Screening of >1000 Pesticides in Fruits and Vegetables

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High-resolution mass spectrometry has provided new opportunities for pesticide analysis in food, feed, and other agricultural commodities. A non-Target Data Acquisition for Target Analysis (nDATA) workflow was developed to screen pesticides in fresh produce based on liquid chromatography full scan MS/variable data independent MSMS (LC-FS MS/vDIA MSMS) acquisition and a 1200 pesticide database. Three laboratories participated in the workflow study to evaluate pesticide identifications in 10 produce matrices and compare laboratory performance by determining false positive and false negative rates in different blank and fortified samples. Pesticides were identified by retention times and mass errors of the precursor and product ions. Incurred residues were evaluated by the nDATA workflow in archived food samples and results were compared to targeted analysis approaches by GC-MS/MS, LC-MS/MS, and LC-FS MS/DDA MSMS. GC-MS/MS identified three pesticides that were not amenable to LC-MS analysis in the produce samples. The nDATA workflow utilizing LC-FS MS/vDIA MSMS and the 1200 pesticide database was able to determine parent pesticides at levels > 10 µg/kg, consistent with LC-MS/MS, GC-MS/MS, and LC-FS MS/DDA MSMS targeted procedures, but was also able to identify metabolites that were not provided in MRM or inclusion lists by the targeted procedures. The nDATA workflow offers a potential screening compliment to targeted LC-MS methods, not only for pesticides, but for other LC-amenable chemical residues and contaminants.

O-25

Capillary electrophoresis, normal atmospheric pressure, and particle beam mass spectrometry for residue applications

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This work addresses the application of capillary electrophoresis (CE)-, normal atmospheric pressure (NAP)-, and particle beam (PB)– mass spectrometry (MS)to a diverse variety of agricultural, ecological, environmental applications. CE-MS has revolutionized the spectrometric analysis of highly polar agrochemicals, including protic organics and inorganics, formerly prone to matrix suppression and chromatographic complications associated with reversed phase and ion exchange techniques. We detail the analysis of a suite of polar pesticides and nutrients of great interest for a number of reasons, including: highly publicized concerns about health effects (i.e. – glyphosate), MRL violations caused by biogenic and anthropogenic interferents (i.e. – fosetyl-aluminum, and phosphonic / phosphoric acids), and the large amounts of these compounds that are used each year to maximize the global food supply. NAP-MS has been directed, and contributed greatly, to our understanding of “fugitive emissions”, particularly in the context of quantifying fumigant fluxes from treated matrices. PB-MS has recently been applied to MOSH/MAOH residues and advantages if this novel approach will be discussed and detailed.

O-26

Cutting Edge Methods for Pesticide Residue Analysis

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Pesticide residue analysis in vegetables is facing the requirement of having to detect more and more compounds in more and more matrices at lower and lower concentrations. These combined effects have led to a situation in which more than one extraction method combined with QqQ technology in necessary but often they present some limitations. It is a fact that mass spectrometry performance has greatly improved over recent decades, especially in terms of detection sensitivity. In general, if the available sensitivity is much higher than the required LOQs, the procedure selected is to inject lower volumes or dilute the food extracts to avoid the negative matrix effects. However, it is a fact that false positive and especially false negative results can be/are reported.
Consequently, new developments are still seeking to diminish the negative impact of interferences when large scope of residue covered in the methods or co-extractives especially within complex matrices. New technologies and analytical tool innovations will play a relevant role in the future for achieving these high exigencies of sample workflows.
From them, we will present new possibilities considering advantages and limitations to obtain an additional degree of selectivity while maintaining the full-scan-based detectability of all the compounds of interest have led to new data-independent acquisition (DIA) techniques by HRMS combining new advances in DDA, SWATH or IMS. Last, but not least, new clean up approaches based on QuEChERS extraction method will be evaluated.

O-27

Significance of Ochratoxin A in Agricultural Commodities and Processed Food Products

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Ochratoxin A (OTA) is a potent mycotoxin targeting mainly the kidney as well as the liver and immune system. This possible human carcinogen occurs frequently in many agricultural crops and their processed foods products around the world due to the diversity of OTA producing fungi and the stability of OTA during food processing. Hence, in addition to the science-based regulations, it is important to maintain surveillance programs including all major cereal grains, beans, coffee, wine, beer, fresh and dried fruits, cocoa, nuts, spices, infant formula and infant cereals. In addition to OTA, formation of degradation products and their residual toxicity should also be considered when assessing the exposure and health risk. The known degradation products include OTα, OTα amide, and 14-*R*-OTA while their relative toxicity is known to be less than that of their parent compound. In detection and quantification of OTA and its analogs, LC-MS/MS has been proven to be the most valuable analytical technique yet. As the food industry continues their effort to reduce OTA through various technologies, including both thermal and non-thermal processing, detection and identification of novel degradation product(s) should also be of a priority.

O-28

Analysis of Ergot Alkaloids in Cereal-based Food Products using LC-MS/MS

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Fungal infection of cereal grains such as rye, wheat, oat, and barley by *Claviceps* species causes the formation of

dark sclerotia that contain different classes of toxic ergot alkaloids (EAs). Despite effective cleaning procedures, the contamination of EAs in cereals and cereal-derived food remains a possibility and reliable analytical methods are needed to simultaneously monitor diverse groups of EAs in different food commodities. In this work, a targeted LC-MS/MS method was developed and single laboratory validated for the simultaneous detection and quantification of six most prominent EAs and their corresponding epimers in cereal grains. Samples were fortified with a surrogate standard and extracted using 85% acetonitrile and 15% (v/v) water containing 200 mg/L ammonium carbonate. Chromatographic separation of epimers was achieved using mobile phases under alkaline condition (pH 9) on a high pH resistant C18 column. Method performance was evaluated for accuracy and precision in six matrices (wheat flour, rye flour, pearl barley, steel cut oats, baby oatmeal, and multi-grain flakes) fortified with 2.5, 10, and 40 µg/kg native standards. Quantification was achieved using matrix calibration standards over the range of 0.5 to 100 µg/kg. Estimated matrix dependent LOQs ranged from 0.3 to 1.4 µg/kg. The majority of target EAs showed recoveries that ranged from 80-120% with relative standard deviations less than 20%. The established workflow provides a selective and sensitive method to simultaneously quantify twelve EAs in a variety of cereal-based food products.

O-29

Development of an LC-MS/MS Method for the Detection of Microcystins in Marine and Estuarine Shellfish

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In recent years, increased attention has focused on the risks from exposure to cyanotoxins, such as microcystins (MCs), through consumption of freshwater seafood. Several reports have shown that these primarily freshwater toxins can also be found at varying concentrations in marine and estuarine shellfish. To better assess the concentrations found in these vectors, and the risks they pose to consumers, a validated analytical method is required. To date, several protocols have been published, but the recovery of the targeted MCs have varied. In addition, many methods based on mass spectrometry suffer from varying degrees of matrix interference. The major difficulty for extraction and accurate detection of MCs is their polarity with the Kow between -1 to ~4. Herein, an extraction and detection protocol were developed for MCs (i.e., MC-LA, -LF, -LR, -LW, -LY, -RR, -YR, and nodularin-R) from Eastern oysters (*Crassostrea virginica*), blue mussels (*Mytilus edulis*), hard clams (*Mercenaria mercenaria*) and softshell clams (*Mya arenaria*). Samples were extracted using 20 mL 80% aqueous methanol acidified with 0.1% formic acid. MCs were analyzed using a Waters Acquity UPLC system with Sciex QTrap mass spectrometer operated in negative ESI mode. The evaluation of the method was based on spike recovery, matrix effects, precision, specificity, and ruggedness. Overall, the recovery ranged from 85 to 117%. Matrix effects were between 90 and 120%. The extraction protocol reduced matrix interference from other compounds and provided cleaner extracts which will benefit the monitoring efforts.

O-30

Development of a Cyanobacterial Dietary Supplement Reference Material for Multiple Classes of Cyanotoxins

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Some cyanobacterial dietary supplements are prepared using biomass harvested from lakes and ponds where multiple species co-occur. This can result in the contamination of products with cyanotoxins that present a risk for the consumer. Reference materials are therefore required to assist in the development, validation and quality control of analytical methods used to assess raw material and for product testing in both research and regulatory environments. Here, we report the development and preliminary characterization of a dietary supplement matrix certified reference material (CRM) for multiple classes of cyanotoxins.
The CRM was prepared by blending biomass of a variety of toxic cyanobacterial strains with a typically-consumed non-toxic strain (*Aphanizomenon* sp.). The freeze-dried, homogenized material was aliquoted into over 2000 identical units and will be characterized for toxin concentration, homogeneity and stability using a novel multi-class extraction and

LC-MS/MS method. A suite of class-specific methods will also be used to assign certified values to toxins for which certified reference material calibration solutions are available, using a combination of isotope dilution and standard addition calibration. These include five microcystins (MCs), eight saxitoxins (STXs), nodularin-R, anatoxin-a and cylindrospermopsin, with preliminary concentrations ranging from 1 to 35 µg/g dry weight. Finally, untargeted high-resolution mass spectrometry methods will be used to characterize the rich profile of over 50 non-certified cyanotoxins and other cyanobacterial secondary metabolites present.

O-31

Harmonizing pesticide tolerances through regulatory capacity building: The CERSA-USDA FAS collaboration

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Pest pressures take a devastating toll on crop yields resulting in loss of food production capacity. The use of pesticides in these systems is critical to allow growers to increase the crop yield while reducing costs of other alternative pest control practices. Maximum Residue Limits (MRLs) for pesticides are the maximum amount of pesticide allowed on the crop at the time of harvest before it goes to market and are specific to each pesticide/crop combination. Individual countries can set their own MRLs and differences among countries can result in trade barriers impacting the decisions of growers in pesticide use and agricultural production practices. Countries without robust regulatory programs may adopt MRL values adversely impacting crops imported into their country, exported to other countries, or create regulatory systems that do not educate farmers and prevent pesticide use practices that could result in MRL violations. This ultimately could impact the pesticides available for use by farmers who would be forced to accept yield losses, ship products with defects in quality, or to use less effective or more costly methods of controlling pests if they cannot use certain key pesticides. This leads to higher production costs and less availability of food commodities. To assist countries without robust programs, the USDA's Foreign Agricultural Service (FAS) has partnered with the Center of Excellence for Regulatory Science in Agriculture (CERSA) to build regulatory capacity of nations within Asia, Latin America, and Africa. The goal of these efforts is to reduce barriers to agricultural trade, increase availability of crop protection products, and educate regulators through workshops and training programs to regulatory groups and assistance with regulatory program development.

O-32

Novel Low Sulfur Distillate and Residual Fuel Oils as Emerging Environmental Contaminants

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The International Maritime Organization (IMO) issued revisions to MARPOL Annex VI to reduce global emissions from marine transport, implementing a low sulfur fuel policy for January 2020. IMO 2020 limits sulfur content of maritime fuel to 0.5% for very low sulfur fuel oils (VLSFO) and 0.1% for ultra-low sulfur fuel oils (ULSFO) worldwide. As a result, LSFO sales in Singapore, the largest global bunkering hub, increased from 174 thousand tons of LSFO in September 2019 to 2,896 thousand tons in September 2020. Additionally, the spill of 300,000 gallons of a residual LSFO from M/V *Wakashio* in July 2020 was the first major spill of this new class of fuels. There is a paucity of chemical and physical data concerning LSFOs, leading to less certainty in oil spill response and post-spill restoration activity. Consequently, there is fear among regulators and responders that these oils are entirely different from conventional fuel oils. The current work conducted a preliminary survey of the physical and chemical chromatographic properties of 18 VLSO and ULSFOs. Results showed the chemical composition of distillate and residue LSFOs are similar to traditional marine fuels. The bulk properties of these oils are also within the expected values of traditional or conventional fuels. Ultimately, the desulfurization of fuel oils does not appear to cause a substantial change in chemical and physical properties relative to pre-IMO 2020 fuels.

O-33

Method Performance using dual WAX/GCB and GCB/WAX SPE Formats for Draft EPA Method 1633

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EPA draft method 1633 covers the extraction and analysis of 40 per- and polyfluoroalkyl substances (PFAS) in a variety of environmentally relevant matrices known as *"Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS".*[1] The method involves a two-step SPE approach using weak anion exchange (WAX) followed by graphitized carbon black (GCB) clean-up in a powder format, known as dispersive GCB (dGCB). For water samples, dGCB is added after extraction, but added before SPE for soil samples. The purpose of the additional GCB clean-up step is to eliminate matrix that can cause interference and reduce bias. Adding dGCB is very labor intensive and therefore not practical due to the added time needed to add, mix, and centrifuge for each sample, especially in high throughput laboratories. For these reasons, cartridges were developed as a single cartridge stacked with WAX and GCB sorbents that function as a traditional Solid Phase Extraction (SPE) cartridge with a built-in polishing step to meet the method guidelines. The goal of this study was to evaluate the method extraction procedure using the dual step approach using WAX plus Dispersive SPE compared to a single approach using a dual stacked SPE phases for wastewater, groundwater and surface water extracts from EPA 1633 validations studies.

O-34

Beyond the traditional targeted analysis approach: Comprehensive screening of PFAS in South Florida environments by Non-Targeted Analysis

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Per- and polyfluoroalkyl substances (PFAS) are a group of anthropogenic pollutants that are found ubiquitously in surface and drinking water supplies. Due to their persistent nature, bioaccumulative potential, and significant adverse health effects associated with low concentrations, they pose a concern for human and environmental exposure. While targeted analysis allows for the accurate identification and quantitation of some legacy and emerging PFAS, the information on thousands of unknown PFAS precursors, degradants, and metabolites is limited by the availability of analytical standards and scientific knowledge on fate and transformation of these contaminants. Non-targeted analysis (NTA) using high-resolution mass spectrometry (HRMS) enables a more comprehensive PFAS characterization in environmental samples. In this study, we have developed and compared NTA workflows based on an online solid phase extraction- liquid chromatography high resolution mass spectrometry (online SPE-LC-HRMS) method followed by data processing using Compound Discoverer and FluoroMatch for the screening of PFAS in drinking waters from populated counties in South Florida, as well as in surface waters from Biscayne Bay, Key west, and Everglades canals. Tap water showed the highest number of PFAS features, indicating a poor removal of these chemicals by water treatment or perhaps the breakdown of PFAS precursors. The high number of PFAS features identified only by CD (206) and FluoroMatch (76) emphasizes the complementary aspects of these data processing methods. Overall, NTA can provide complementary information on PFAS species in the environmental samples, which is needed to better evaluate their toxicological and potential impacts.

POSTER ABSTRACTS

P-01

Assessment of Per- and Polyfluoroalkyl Substances (PFAS) in Tap Waters from Miami-Dade, South Florida

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Per- and poly-fluoroalkyl substances (PFAS) are a class of synthetic chemical compounds widely used in various industrial and commercial products due to their unique properties, including resistance to heat, water, and oil, which make them useful in a wide range of applications, such as non-stick cookware, water-resistant textiles, and firefighting foams. However, their persistence in the environment and the human body has led to concerns about their potential health effects. Exposure to PFAS has been associated with various adverse health outcomes, including developmental delays, reduced immune system function, and an increased risk of certain cancers. One of the most concerning routes of exposure to PFAS is through drinking water contaminated with these chemicals. Drinking water contaminated with PFAS has become a growing public health concern in recent years, and there is a need for more data on the prevalence of these chemicals in different regions. To address this concern, this study aims to determine the levels of PFAS in 40 tap water samples collected between March and April 2023 (dry season) from nineteen zip code areas in Miami Dade County, South Florida. The samples were processed using Solid Phase Extraction (SPE) followed by liquid chromatography-tandem mass spectrometry (LC-MS/MS), a sensitive and reliable method for analyzing PFAS in water samples. The results of this study will provide valuable information on the extent of PFAS contamination in drinking water sources in Miami-Dade County and adds to the growing evidence on the prevalence of PFAS contamination in some regions of the county.

P-02

Target analysis of PFAS in surface water from Biscayne Bay canals

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It is well established that per- and poly- fluoroalkyl substances, (PFAS) are present in the environment as “forever chemicals” due to their stable, persistent, and accumulative properties. While some PFAS, such perfluorooctanesulfonic acid, (PFOS) and perfluorooctanoic acid, (PFOA), were phased out in the U.S., new alternatives are being produced and these shorter-chain PFAS can be as persistent and toxic as the previously banned compounds. Therefore, there is a need to monitor levels of PFAS in the environment to understand their effects on humans and aquatic organisms. Anthropogenic input is leading to raised levels of contaminants, such as PFAS, in the Biscayne Bay. These contaminants and excess nutrients are leading to fish kills and seagrass die off, which is a major concern in the local community. There is still a knowledge gap in regard to the fate and transport of these compounds in South Florida. While it is known that anthropogenic sources are discharging along Miami River, Little River, and Biscayne Canal, levels along the canals and specific point sources have no been investigated in detail. In this study, we have initially collected 15 surface water samples from Biscayne Canal 8 in April 2023 for targeted analysis of 40 PFAS following EPA Method 1633. A sensitive and robust method for the detection and identification of legacy and emerging PFAS will be developed and optimized using solid phase extraction, (SPE) followed by liquid chromatography triple quadrupole mass spectrometry, (LC-QQQ-MS).

P-03

Occurrence of per- and polyfluoroalkyls substances (PFAS) in groundwater from Miami-Dade, South Florida

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PFAS were first synthesized 80 years ago and are still part of consumer products, non-stick pans, and aqueous firefighting foams (AFFF). These compounds are water soluble and resistant to biodegradation due to the exceptional stability of C-F bonds. Their inability to be removed by traditional water treatments and the health risks associated with exposure to PFAS have brought the need to understand better its fate and transport in the environment. Once these compounds are released into the environment, it enters the water cycle and follows different transport mechanisms toward the groundwater. Biscayne Aquifer is one of the most productive aquifers in the world. Its infiltration capacity is fast, facilitated by conduits and sinkholes, making it prone to contamination. This study assessed the occurrence and composition of legacy and emergent PFAS in monitoring wells in the Miami-Dade area, South Florida. For this purpose, 15 groundwater and 7 point-of-exit samples were taken in 6 locations in Miami-Dade County during the dry season in April 2022 (N=21). The methodology involved the preconcentration of 250 mL of groundwater by solid phase extraction process (SPE) using a weak anion exchange (WAX) cartridge, followed by liquid chromatography-tandem mass spectrometry (LC-MS/MS). The method was validated in terms of linearity, detection limit, precision, and accuracy. Perfluorooctanoic sulfonate (PFOS) was detected in all the collected samples at concentrations above the EPA's proposed Maximum Contaminant Levels (MCLs) of 4 ppt. PFOS, PFOA, and PFBA were the most frequent and abundant compounds, and Miami-Springs wells exhibited the highest total PFAS concentration.

P-04

Capillary electrophoresis, normal atmospheric pressure, and particle beam mass spectrometry for residue applications

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This work addresses the application of Capillary electrophoresis (CE), normal atmospheric pressure (NAP), and particle beam (PM) – mass spectrometry (MS) to a diverse variety of agricultural, ecological, environmental applications. CE-MS has revolutionized the spectrometric analysis of highly polar agrochemicals, including protiic organics and inorganics, formerly prone to matrix suppression and chromatic complications associated with reverse phase and ion exchange techniques. We detail the analysis of a suite of polar pesticides and nutrient of great interest for several reasons, including highly publicized concerns about health effects (i.e.-glyphosate), MRL violations caused by biogenic and anthropogenic interferents (i.e., fosetyl-aluminum and phosphonic / phosphoric acids), and the large amounts of these compounds that are used each year to maximize the global food supply. NAP-MS has been directed, and contributed greatly, to our understanding of "fugitive emissions", particularly in the context of quantifying fumigant fluxes from treated matrices. PB-MS has recently been applied to MOSH/MOAH residues and advantages if this novel approach will be discussed and detailed.

P-05

Effect of substrate porosity in the analysis of residues using Surface Enhanced Raman Spectroscopy (SERS)

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Agrochemicals are engineered to destroy insects, weeds, and fungi that could spoil crop yields. They are toxic by design, and at levels above maximum residue limits, they pose risks to human health and the environment. In order to monitor these levels, the products need to be tested routinely. Surface Enhanced Raman Spectroscopy (SERS) is an analytical technique suited for field-based chemical analysis with molecular level detection capability. Paper-based SERS substrates are often preferred for their surface roughness (3D structure), disposability, and use as swabs. In this presentation we will show that paper-based silver SERS substrates, prepared using the Print-Expose-Develop method, result in a loss of signal owing to two primary reasons. Firstly, the interaction between the target molecules which are present across the paper depth, and the top nanostructure layer, is limited. Secondly, the sub-micron depth of field in typical spectroscopic systems limits the signal collection from the nanostructures and molecules across the cross-section of the paper substrate (~100 µm thick). To overcome this, we prepared silver dendritic structures on non-porous metal foil via galvanic displacement. These substrates are easy to prepare and show good reproducibility, RSD~0.10. For the detection of malachite green dye (antimicrobial in aquaculture), the most intense peak can be resolved at concentrations as low as 1 nM. Here, results from studies for detection of thiabendazole (a fungicide), separation studies of mixture of dyes, and the signal collection efficiencies of both types of substrates will be compared in the context of field-detection using a portable Raman Spectrometer.

P-11

Various Environmental Residue Testing within the Colorado Department of Agriculture Biochemistry Lab

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The Colorado Department of Agriculture Biochemistry Lab's Pesticide Unit performs extensive environmental residue testing. Three areas of focus are the analysis of hemp for d9-Tetrahydrocannibidiol, agricultural water monitoring for trace nutrients and pesticides, as well as residual pesticide enforcement sampling of vegetation, soils, bees, and investigations involving human health. Our laboratory has made improvements to each of these testing programs to improve data quality and expand our scope of analysis. In our hemp analysis we have updated the extraction process and converted the GC-FID to operate using Hydrogen carrier gas instead of Helium in response to the nationwide shortage. The agricultural water monitoring program has been able to expand testing capabilities to include SEAL nutrient analyses and additional pesticide residues. The pesticide residue enforcement program has done extensive testing to eliminate the use of QuEChERS, vastly improving analyte recovery and reproducibility in all matrices.

P-12

Characterization of Homogeneity in Mycotoxin Samples Using Laser Diffraction Particle Size Analysis

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Sample homogeneity dictates whether analyzing a test portion of an entire sample can provide representative information about incurred mycotoxins. In this study, we evaluated homogeneity using laser diffraction particle size analysis and International Organization for Standardization (ISO) Guide 35: 2017. Incurred whole corn, compound feed, peanut butter, and wheat flour (500 g each) were homogenized using wet, cryogenic, or dry milling. We used a sample dividing (riffling) device to obtain representative subsamples (25 g each) and developed a laser diffraction particle size analysis procedure by optimizing key parameters such as refractive index, absorption, and stirring rate. Homogeneity of matrix subsamples was characterized using the optimized laser diffraction procedure. An assessment of homogeneity was also performed for individual mycotoxins in each incurred matrix sample following the procedure described in ISO Guide 35. Concentrations of incurred mycotoxins were determined using liquid chromatography-mass spectrometry (LC-MS). Within- and between-sample variabilities of incurred aflatoxin B1 in peanut butter; deoxynivalenol in corn, compound feed, and wheat flour; and fumonisins in compound feed corroborated that when particle size measurements were less than 850 µm, mycotoxin concentrations were statistically consistent across independent test portions (1 g each). In the context of this work, findings were independent of milling method, matrix commodity, or mycotoxin. This study highlights the benefits of laser diffraction particle size analysis and suggests use as a test procedure to evaluate homogeneity in new sample commodities.

P-13

Investigating Chemical Interactions of PFAS for the Development of Efficient Extraction Methodologies and Estimation of PFAS Toxicity using the Software Percepta

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Per and polyfluoroalkyl (PFAS) are chemicals with properties that effectively repel water and oil. Its use has led to its presence in various environments, including water, soil and biota. Methodologies to extract different PFAS matrices are being studied due to the wide range of physicochemical properties of these compounds. This study used the Percepta software to predict their intermolecular interactions and associated toxicity from the physicochemical properties of 476 PFAS (PFAS Master List EPA Database). The physical-chemical properties pKa, Log P and Log S were used to determine the best chemical interaction for extraction of each PFAS group. Among the results found, it was observed that mixed-mode polymeric sorbents provide greater selectivity and sensitivity to extract acidic compounds with anion exchange groups. The software also predicted the LC50, a measure of acute toxicity, for 476 PFAS in water fleas and fatty minnows. The results showed that the PFAS studied have the potential to exert mild toxicity depending on the organism, with LC50 values ranging from 50-100 mg/L for water flea and 25-50 mg/L for goldfish. In conclusion, predicting physicochemical properties and clustering of PFAS using Percepta software can improve understanding of PFAS chemical interactions.

P-14

Tree Nut Method Validation and Matrix Extension Utilizing QuEChERS and Select Pesticides

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Paclobutrazol (PBZ), a plant growth regulator, is currently being used in Mexico other countries to achieve higher yields of nuts. This is achieved by stunting the growth vertically and promoting shoots and stems to grow more horizontally producing more sites for tree nut growth. PBZ has no established tolerances for food commodities in the US and impacts to human health are still unknown which led to the development of this study. Pecans are a majority commodity produced in Georgia, and a matrix extension was seen as a vital aspect of this project for protection of this state product. Three tree nut commodities (pecans, walnuts, and almonds) were surveilled for the presence of paclobutrazol, and other pesticides previously established in the QuEChERS food method. All three tree nut commodities were sampled from both domestic and non-domestic sources available at retail food establishments. An addition of the commonly used pesticides served as a matrix extension for the already ISO 17025 accredited method. This method utilizes the QuEChERS for prep

and samples were analyzed using a Q-trap 5500 LC-MS/MS. 5-point standard curves were calculated for each tree nut for PBZ and other pesticides. Numerous calculations were performed to measure performance of the analysis: relative standard deviations, limit of detections, limit of quantification, % recoveries, R^2 values, measurement of uncertainties, control charts, Anova tests, and raw compiled data. Overall, the data indicated that the method validation for PBZ and the matrix extension of tree nuts utilizing QuEChERs was successful.

P-15

Assessment of per- and polyfluoroalkyl substances in two species of captive delphinids from the United States

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Delphinids are long-lived top predators that serve as important sentinel species for the health of the environment they live in. Due to the bioaccumulation and biomagnification processes, delphinids may be exposed to contaminants like per- and polyfluoroalkyl substances (PFAS), which are concerning contaminants due to their wide use in commercial and consumer products. Thus, we investigated the contamination of 30 PFAS in 85 blood samples from two species of captive delphinids at different life stages (*Tursiops truncatus* [n = 41; 14 individuals], and *Orcinus orca* [n = 44; 14 individuals]) located across three SeaWorld Parks in USA, from 1994 to 2020. Blood samples were collected from individuals' central fluke vein and serum was separated by centrifugation and stored at -80°C. Samples were extracted by adding methanol and vortexing. Extracts underwent evaporation, cleanup, and filtering processes and were analyzed by LC-MS/MS. All PFAS were detected in at least one of the individuals. Higher concentrations of PFOS (*T. truncatus*: 2482 $ng.ml^{-1}$; *O. orca*: 406.9 $ng.ml^{-1}$) and PFNA (*T. truncatus*: 246.2 $ng.ml^{-1}$; *O. orca*: 137.0 $ng.ml^{-1}$) were observed in both species. Higher mean ΣPFAS was found in *O. orca* calves (32%) and in *T. truncatus* immature males (38%). Mother-calf PFAS transference was also observed in both species, indicating transference via placenta and lactation. Associations between PFAS and stress biomarkers are also being investigated. Continued PFAS monitoring in delphinids is essential to better understand the variations of these contaminants across space, time, demographic and reproductive stages, as well as their adverse health effects.

P-16

Persistence of Chlorpyrifos and Its Metabolites on Field-grown Vegetables

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In a randomized complete block design 18 field plots were separated using metal borders. Plots were planted with kale and collard greens 45 d seedlings, and drip irrigated as needed. The plants were sprayed with Lorsban 4E (44.9% A.I.) that contained 425 g A.I. as a foliar spray $acre^{-1}$. Leaves, collected at different time intervals of 1 hour, 1, 5, 7, 12, 14, and 23 d following spraying, were blended with ethyl acetate. Concentrated extracts were cleaned up using open glass chromatographic columns (20 × 1.5 cm) packed with Florisil. A fraction of the concentrated extract was added to the column and eluted with 100 mL of a mixture of ethyl acetate: and petroleum ether (30:70 v/v). Residues of chlorpyrifos, chlorpyrifos oxon, and 3, 5, 6-trichloro-2-pyridinyl (TCP) were determined using a gas chromatograph (GC) equipped with an electron capture detector (ECD) and confirmed using a GC equipped with an MSD. Chlorpyrifos residues on collard leaves varied from about 14.6 $\mu g\ g^{-1}$ at the time of application to 7.9 $\mu g\ g^{-1}$ after one d to about 5 $\mu g\ g^{-1}$ after 7 d and to 0.5 $\mu g\ g^{-1}$ after 23 d. Whereas the oxon analog was a small proportion of the parent chlorpyrifos. TCP residues on collard fluctuated during the growing season which could be due to the fact, that both chlorpyrifos and the oxon are hydrolyzed to TCP. This dissipation pattern revealed that a chlorpyrifos half-life value ($T_{1/2}$) of 5.1 d was found on the collard, whereas a $T_{1/2}$ of 2.2 d was found on kale.

P-17

NIST's Recent Developments in Measurement Services for Food Safety

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The food safety program at the National Institute of Standards and Technology (NIST) provides measurement services, including reference materials (RMs) and quality assurance programs (QAPs), to support food safety measurements. Ongoing research interests for contaminants in food include pesticide residues, per-and polyfluoralkyl substances (PFAS), and mycotoxins. NIST recently released a two-level glyphosate in oat flour suite, RM 8238 and RM 8239, with nominal mass fractions of incurred glyphosate at 450 ng/g and 70 ng/g, respectively. NIST is examining the feasibility of producing small batch research grade test materials of food matrices spiked with pesticides. The first protype material consists of 11 pesticides in spinach that was prepared by cryohomogenization. Stability testing of this material is currently underway. NIST is also producing RMs with incurred PFAS in a number of matrices including bovine tissue, porcine tissue, fish tissue, spinach, corn silage, milk and eggs. These candidate RMs were prepared by using matrices that were suspected of having detectable levels of PFAS and were screened prior to packaging. A RM of incurred mycotoxins in animal feed is also being developed. QAPs are another measurement service provided by NIST. These interlaboratory study programs are designed to help participants identify and understand community wide measurement challenges and improve the accuracy of their measurements. Recent studies of the Food Nutrition and Safety Measurements QAP (FNSQAP) include toxic elements in baby foods and chocolate, acrylamide in chocolate and coffee, glyphosate in food, and phthalates in infant formula and cheese.

P-18

Quick Method for The Analysis of Highly Polar Pesticide (Glyphosate) in Fruits and Vegetables

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In food production, pesticides are essential for extending shelf life and enhancing the trade of high-quality foods. Despite this, chronic exposure to these chemicals may adversely affect human health. N-(phosphonomethyl) glycine or Glyphosate is the most widely used chemical herbicide. Glyphosate is classified as a high polar pesticide (HPP).This study aimed to determine the glyphosate residues in fruits and vegetables. The cucumber was selected as a representative crop for analyzing Glyphosate in fruits and vegetables for method validation based on the Quick Method for the Analysis of Highly Polar Pesticides (QuPPe-PO-Method) using Shimadzu LC-MS/MS instrument fitted with a Torus DEA column. Glyphosate validation was conducted at three levels, 20, 50, and 100 µg/kg, and the limit of quantification (LOQ) was 20 µg/kg. There is satisfactory linearity in the calibration curves, with a coefficient of determination (R2) > 0.99. Regarding repeatability, the recovery values range from 89.6 to 93.6%, with an RSD from 4.04 to 10.27%. In conclusion, the validation provided the expected performance regarding scope, specificity, accuracy, sensitivity, repeatability, and reproducibility.

P-19

Proportionality between spraying amounts of pesticides in crop fields and their initial deposit amounts on surfaces of watermelon and muskmelon

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The proportionality between application rates of pesticides and their residue levels is useful for comparing the deposit ability of pesticides on different types of fruit surfaces such as major and minor crops. We evaluated the proportionality on residue data of muskmelon with rough-ridged surface and watermelon with flat-smooth surface from the supervised crop field trials. Because of eliminate the effect of pesticide degradation and/or loss, residue data of pre-harvest interval after the final application of 1 day were selected for this study. Statistical analysis of all residue data for muskmelon (n=172) indicated a strong correlation between the single application amounts of pesticides and their deposit amounts. On the other hand, statistical analysis of all residue data for watermelon (n=148) indicated a poor correlation. Except for polar pesticides, there was a good correlation on the residue data for watermelon (log P_{OW} > 4, n=77). The difference in deposit amounts of pesticides on the two types of fruit surfaces estimated approximately 3 times (muskmelon / watermelon).

P-20

Fluorometric determination of unsulfonated aromatic amines in certified color additives using fluorescamine derivatization

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Color additives must be pre-approved and listed in the Code of Federal Regulations (CFR) before they may be used in specific product categories regulated by the U.S. Food and Drug Administration (FDA). In addition, the color additives listed in 21 CFR parts 74 and 82 must be batch certified by the FDA to ensure compliance with purity requirements. FDA's Color Certification Program currently uses ultra-high-performance liquid chromatography (UHPLC) with photodiode array detection (LC-PDA) or tandem quadrupole mass spectrometry detection (LC-MS/MS) to determine trace-level unsulfonated aromatic amines in certifiable color additives. We have developed and validated a new method using UHPLC with fluorescence detection (LC-FLD) for the determination of aniline (ANL) in D&C Red No. 17 and *p*-toluidine (*p*T) in D&C Green No. 6, D&C Violet No. 2, and Ext. D&C Violet No. 2. Derivatization of the impurities with fluorescamine (a nucleophilic substitution reaction in aqueous medium) produced fluorescent analytes (390 nm excitation, 475 nm emission) capable of sensitive and selective quantitation. We applied the new LC-FLD method to surveys of samples of color additives from domestic and foreign manufacturers requesting certification within the last three years. We compared the LC-FLD method to our current LC-PDA and LC-MS/MS methods with respect to suitability of multiple detectors, performance, sample preparation, and cost. We evaluated the LC-FLD method for sensitivity, reproducibility, accuracy, and recovery. Our data indicate that the limits of detection (0.0003%) and quantitation (0.001%) are well-suited for the determination of ANL or *p*T, which have specifications of up to 0.2%.

P-21

LC-MS/MS method development for potentially "new" contaminants in food matrices of plant and animal origin

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This study was carried out in cooperation with the European Food Safety Authority (EFSA), which requested the development of methods for compounds potentially contaminating 15 different unprocessed plant or animal origin food matrices. Very polar compounds (n = 20) were selected based on a previous study handled by EFSA. Method development included both an extraction step and an analytical separation and detection. Commonly used procedure for the determination of highly polar compounds, based on extraction with 50% aqueous methanolic solution (QuPPe), was used for sample extraction. Liquid chromatography using hydrophilic interactions (HILIC) coupled with tandem mass spectrometry was used for the analysis of selected compounds. The main indicators of the developed analytical method suitability for the analysis of selected compounds in the given matrices were recovery, which was higher than 70 %, and repeatability, which was lower than 20 %, both for most analyte-matrix combinations. This study also included screening of selected compounds in real samples of various unprocessed plant and animal foods. Among the most detected compounds in the analyzed samples were: 1-vinyl-2-pyrrolidone, N,N-dimethylacetamide, N-methylacetamide and N-methyl-2-pyrrolidone. Matrices with the widest spectrum of detected compounds were flour or herring, on the other hand, none of the analyzed compounds were detected in kale, peas and strawberries. This study brings new analytical data for newly monitored compounds and their findings in unprocessed foods.

P-22

Chili peppers and chili powders: any chemical safety issues of concern?

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Chili peppers and products thereof are widely used in cuisines around the world. However, food safety issues, particularly an unauthorized and excessive pesticide residue levels in imported fresh chili peppers and chili powders, are of concern. Moreover, the presence of mycotoxins in chili powder might be of concern.
In this study, the occurrence of pesticide residues, their metabolites and mycotoxins in a set of fresh chili peppers and chili powders was investigated. For the determination of altogether 417 pesticide residues and mycotoxins, UHPLC-MS/MS was used. Moreover, pesticide metabolites were screened using UHPLC-HRMS/MS. The obtained results were assessed against the Maximum Residue Levels (Regulation (EC) No. 396/2005 – pesticide residues) and Maximum Levels (Commission Regulation (EC) No. 1881/2006 - mycotoxins). In fresh chili peppers and chili powders, 50 and 61 different pesticide residues, respectively, were detected, including carbofuran and chlorpyrifos that are banned in European Union. In case of four fresh chili and three chili powder samples, the maximum residue level violation was found. Also, in two of three chili powders declared as ´organic´, tolerable level of residues, 0.01 mg/kg, was exceeded. Furthermore, various metabolites (e.g., desnitro-imidacloprid, spirotetramat-enol-glucoside) of parent pesticides were detected in both, fresh chili pepper and chili powder samples. Mycotoxins were detected only in chili powders. One sample contained ochratoxin A in concentration above the Maximum Levels.

P-23

LC-HRMS determination and identification of antibiotic residues in fruit

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Antibiotic residues are usually associated with foods of animal origin but may also occur in plant products. For example, citrus groves could be treated with antibiotics to combat citrus greening disease caused by bacteria carried by insects. Other antibiotics may be used as fungicides. The goal of this work was to develop a multi-analyte LC-HRMS method for antibiotic residues in fruit. The analytes of interest (streptomycin, kasugamycin, penicillin, oxytetracycline) are challenging due to differences in their polarity and poor stability. Using a procedure based on the EU method for polar pesticides, fruit samples were extracted with acidic methanol. Chromatographic separation was optimized for a HILIC column. A Q-Exactive HRMS instrument with non-targeted data acquisition was used which will allow for detection

of additional residues. Targeted data acquisition was also acquired to obtain more diagnostic product ion spectra for analyte identification. Quantification was performed with matrix-extracted calibration curves and internal standard correction. The extracted curves were linear across a wide range of concentrations (4-800 ppb), and fortified samples demonstrated acceptable recoveries (73-109%) and reproducibility (3-12% RSDs) for most residues in oranges, apples, and cherries at the levels of concern. The response for kasugamycin was more variable and may only be suitable for qualitative screening. This multi-analyte method for antibiotic residues in fruit products is intended to fill a gap in current regulatory monitoring programs.

P-24

Analysis of isoeugenol in aquaculture products using HS-SPME-GC-MS

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Isoeugenol, an active ingredient in fish sedatives, is monitored by the FDA primarily using a solvent extraction and analysis by liquid chromatography-mass spectrometry (LC-MS). Headspace solid-phase microextraction coupled to gas chromatography-mass spectrometry (HS-SPME-GC-MS) offers an alternative analysis method for isoeugenol that avoids the use of organic solvents, is less susceptible to matrix effects, and can be fully automated. SPME has been demonstrated for a wide range of analytes, including volatile phenols, making it appropriate for isoeugenol extraction. Although not an exhaustive extraction technique, SPME can be used for trace-level, quantitative analyses when coupled with mass spectrometry. This work focuses developing and evaluating a HS-SPME-GC-MS for isoeugenol in aquaculture samples. Because of isoeugenol's relatively low volatility, the SPME fiber coating and extraction time impacted extraction efficiency. Using this method, isoeugenol limits of detection in multiple fish matrices (shrimp, tilapia, salmon) were in the low ppb range, below the target testing level (200 ppb). Additionally, by adding deuterated eugenol as an internal standard, good linearity was achieved for calibration curves (R^2 > 95%).

P-25

A Food Poisoning Investigation – The Determination of Aconitine in Edible Spices by LC-MS/MS

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Aconitum is a genus of more than 250 flowering plant species that are native to mountainous regions in the Northern Hemisphere. Alternately known as monkshood, wolfs-bane or aconite, species are cultivated as ornamental plants and with some species, the plant roots are processed and used in traditional herbal medicine. However, many *Aconitum* species are extremely poisonous, containing an alkaloid toxin called aconitine. Symptoms of aconitine intoxication can include nausea, vomiting, dizziness, weakness, irregular heartbeat, and even death from respiratory paralysis or cardiac arrest. Cases of poisoning due to accidental or intentional exposure to aconitine are reported in the literature. Unfortunately, *Aconitum* roots are similar in appearance to those of other plants. Especially in powdered form, *Aconitum* roots can be confused with edible roots that are used as culinary spices, such as *Kampheria galanga* ("sand ginger") with negative consequences. Described here is the development and application of an analytical method for the extraction and determination of aconitine in spices by tandem mass spectrometry coupled with liquid chromatography. Method performance for aconitine and two related toxins, hypaconitine and mesaconatine was demonstrated. The method was rapidly developed and implemented to support an investigation of food poisoning cases where edible spices were suspected to be contaminated with aconitine.

P-26

Suspect screening strategy for pesticide application history based on characteristics of trace metabolites

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Pesticides are used to protect crops against various insects, weeds, and fungi and to increase the yield of the field. Over 1,000 different pesticides are used around the world. They are particularly important in countries that suffer food shortages and continuous population growth. Following their application, pesticides may remain on plants and in the environment or further produce transformation products (TPs) and various conjugates of unknown toxicity, stability, and bioaccumulation properties, posing potential health risks for human beings. Since there are no available reference standards and relevant information, the identification and quantification of pesticide TPs can be a daunting challenge, thus underestimating the risks of pesticides to human health and the environment. In this study, Suspect screening based on high-resolution mass spectrometry technology was regarded as a successful and reliable tool for discovering pesticide TPs in tea plants. 142 TPs from 20 pesticides in fresh tea leaves were identified, of which 16 were sugar conjugates, 12 metabolites were the first to demonstrate in this study. Based on the level system reported by Schymanski et al and the parameter requirements of MS and MS/MS, four confidence levels were then assigned to plausible TPs. 15 metabolites were verified by reference standards and were therefore assigned to the highest confidence level 1. Finally, we analyzed 85 organic tea samples to determine the residual levels of pesticide TPs. And the detection of TPs was used as evidence of pesticides application. These results were indicative of illegal or misuse of pesticides in organic farming.

P-27

Digging through the dirt: the quest for quantifying residues of a complex herbicide in soil and sediment

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Laboratories have a key role to play in supporting sustainable agriculture as analytical work can produce significant quantities of organic solvent and plastic waste. Therefore, adopting advanced LC-MS/MS techniques, such as microflow LC, would reduce solvent waste. Additionally, microflow LC could increase sensitivity and robustness of an analytical method, which are critical factors for global registration and enforcement.

This poster will describe a case study comparing the performance of conventional flow LC-MS/MS to that of microflow LC-MS/MS using a new active ingredient. Optimization of analytical method parameters will be presented along with method validation results including dynamic range, accuracy, precision, and selectivity to support its use in terrestrial field dissipation (soil) studies.

P-28

9-1-1 What's your Soil Emergency? Delivering a modernized, high-throughput method through cross-functional collaboration

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Bayer CropScience has developed a resistance breaking new pesticide with a novel mode of action. A crucial step in the registration of this new crop protection product to the EPA is the execution of terrestrial field dissipation (soil) studies which are used to characterize environmental exposures. This requires development of an analytical method capable of quantifying trace levels (below 10 ng/g) of the pesticide and its metabolites in soil and sediment. Developing a method for this new pesticide and its structurally similar metabolites in a complex matrix such as soil presented some unique challenges. This poster will highlight the strategies used to develop a more modern, sustainable, high-throughput analytical soil method as well as experiments carried out by a cross-functional team to overcome complex challenges which lead to a robust analytical method.

P-29

Validation of a 5-minute assay for the quantitative analysis of mycotoxin residues over a comprehensive range of agricultural commodities

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Natural toxins such as mycotoxins require active monitoring to ensure grain safety and to meet trade regulations. A well-established analytical laboratory method database exists that performs mycotoxin residue quantification, such as HPLC-FLD and LC-MS/MS. On the other hand, agricultural harvesting stations such as grain elevators need more expertise and infrastructure to house such complex instrumentation when several samples need to be certified for immediate cargo release. In this poster, we describe the validation of a 5-minute field test that uses an optimized extraction system for the simultaneous extraction of aflatoxins, deoxynivalenol, fumonisins, ochratoxin A, and zearalenone combined with quantitative immunoassay technology designed to increase sample throughput for the routine residue monitoring of these mycotoxins over a comprehensive range of agricultural commodities. Quantitative results demonstrated the accuracy and robustness of this new field-deployable assay, providing reliable mycotoxin residue analysis and time-saving for grain testing points. Average extraction efficiencies were greater than 70%, accuracies between 80-100%, and CV less than 15%. Standard curves were linear over a minimum range from 2-3000 parts per billion, and r-squared values were greater than 0.98 in all cases.

P-30

Determination of Multi-Class Veterinary Drug Residues in Shrimps by LC-MS/MS

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The method for the determination of multi-class veterinary drug residues in shrimp meat using LC-MS/MS detection was developed and validated. This method allows for fast screening and confirming 38 veterinary drugs in shrimps that are regulated in North America. These targeted drug residues can be categorized into various chemical classes including β-lactams, sulfonamides and derivatives, tetracyclines, fluoroquinolones, quinolones, phenicols, macrolides and benzimidazoles. The detection limits range from 0.05ng/g (chloramphenicol) to 10ng/g (tetracyclines) depending on

different classes.
The procedure involves the extraction of the analytes in the shrimp samples using acetonitrile mixed with water, which is further cleaned up by hexane wash and dispersive C18. The extracts are evaporated to a low volume, made up to the final volume with water, and analyzed by LC-MS/MS. The total running time of LC-MS/MS analysis is within 17 minutes and all the compounds are separated with good peak shapes. The method was validated in the raw white shrimp matrix by spiking 4 different concentration levels, and each level was repeated 6 times. The recoveries of the target analytes are in the range of 75-125%. The precision, expressed as the relative standard deviation (RSD), is in the range of 3-19%. The method ruggedness and selectivity were also studied. The overall results indicate this straightforward, robust and efficient method is suitable to simultaneously detect a wide range of regulated drug residues in shrimps.

P-32

Illegal pesticide residues in food: Observed trends in detections and concentrations for different food groups

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Nestle Quality Assurance Center (NQAC) quantitatively screens, by LCMSMS and GCMSMS, for over 400 pesticide residues in food, herbs, spices and raw materials from all over the United States and parts of the world. A list was compiled and trends were established of the illegal pesticide residues detected, as determined by Title 40, Chapter 1, Part 180 of the Code of Federal Regulations, in different matrix groups for food samples submitted in the 2021 and 2022 calendar years. A range of concentrations was determined for each illegal pesticide residue.

P-33

LC-MS/MS Analysis of Pyrrolizidine Alkaloids in Dietary Supplements Containing Oil Ingredients

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Pyrrolizidine alkaloids (PA) are a group of natural plant toxins with genotoxic and carcinogenic effects to humans that are ubiquitous in nature. Considering potential risk to consumers, maximum levels have been set by the European Commission in Regulation 2020/2040 for a number of commodities including dietary supplements, tea, herbal infusions, herbs, and spices. The extraction protocols used in many of currently available methods may struggle with the effective extraction of oily matrices, including botanical dietary supplements marketed as gelcaps or softgels. In this study, an LC-MS/MS method based on BfR (German Federal Institute for Risk Assessment) assay has been optimized and validated to allow sensitive, selective, accurate and reproducible determination of pyrrolizidine alkaloids in oil samples and dietary supplements containing oils as ingredients. The analyte scope of the method covers compounds specifically mentioned in European Union regulation, allows determination of individual analytes down to 10 µg/kg and report down to the applicable maximum limit of 400 µg/kg (lower-bound sum for total PA content). Data obtained in optimization of the extraction and SPE clean-up procedure will be presented. Method accuracy and precision will be demonstrated based on spiking experiments at multiple levels and compared to acceptance criteria of recovery within 70-120% and RSDs ≤20%. Additionally, results obtained for a limited set of supplement samples sourced from U.S. retail market will be shown.

P-34

Determination of Residual Pyridine and Triethylamine in Dietary Supplements by Direct Injection GC-MS

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Levels of residual solvents represent an important quality and safety attribute of dietary supplements and ingredients. Solvents utilized for extraction of active ingredients may not be completely removed during the manufacturing processes. Analysis of residual solvents is therefore required to allow their monitoring. The United States Pharmacopeia provides limits for more than 60 compounds that are divided into three classes based on assessment of risk to human health. In this study, we focused on development and validation of a direct injection GC-MS assay for the determination of two difficult analytes, pyridine and triethylamine. Considering volatility and basic nature of these analytes, a highly inert amine-specific (CP-Volamine) capillary column has been used and allowed for excellent peak shape. Samples were extracted with acetonitrile; stable-isotope labeled analogs of target analytes were employed in quantification. Acceptable recoveries within 70-120% and RSDs ≤20% were obtained in a wide range of (botanical) dietary supplements representing various dosage forms, as well as botanical ingredients. Good selectivity of the method was demonstrated at concentrations at or below USP limits set at 200 mg/kg and 5,000 mg/kg for pyridine and triethylamine, respectively. The validated LOQ of the assay was 200 ppm for each target analyte.

P-35

Identification of Short Chain PFAS in Infant Formula Using LC-MS/MS and LC-HRMS

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Per- and polyfluoroalkyl substances (PFASs) are a wide group of toxic and persistent anthropogenic chemicals that have become global environmental pollutants. Besides the environment, food chain represents another source of exposure and the risk to consumers related to the presence of PFAS in foods has recently become of increased interest. In this study, we focused on LC-MS analysis and identification of perfluorobutanoic acid (PFBA) and perfluoropentanoic acid (PFPeA) in infant formula matrix. Both of these short-chain compounds yield only a single fragment ion, which limits the confidence in identification of potential positive detections. Samples were prepared using our internal modification of U.S. FDA method C-010-02 for food based on QuEChERS extraction followed by a dispersive SPE cleanup and concentration step. The evaluated analysis workflow included the initial sample screening using triple quadrupole mass spectrometer allowing for a prospective detection of PFBA or PFPeA based on retention time and response of the one available precursor to product transition. In the next step, re-analysis of the suspect samples was performed employing a high-resolution instrument equipped with quadrupole-orbital ion trap mass analyzer to allow for high confidence identification based on accurate mass measurement, as well as for quantification of identified hits. Optimization efforts of the secondary HRMS method to be both fast and accurate will be summarized. We will show several examples of both confirmed and false positive PFAS detections obtained with triple quadrupole instrument along with HRMS confirmatory data collected using HRMS detector to demonstrate validity of this approach.

P-37

The Analysis of 85 Pesticides Commonly Found in Cannabis and Cannabis Related Products by LC-MS and GC-MS

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Residual pesticides in cannabis and cannabis products can pose a major health risk to consumers in both recreational and medicinal applications. The main source of these chemicals comes during the growing process where pesticides are applied to control pest growth. Some of those pesticides, such as organophosphates, are known for their toxic effects not only on human but also wildlife. As a result, regulators and standardization bodies such as ASTM and AOAC has published different test methods to analyze residual pesticides in cannabis and cannabis related products. AccuStandard was able to prepare, identify and quantify over 80 pesticides commonly used during cannabis farming. The analysis was conducted using multiple Certified Reference Materials (CRMs) prepared by AccuStandard and formulated based on the compatibility and the stability of those chemicals. Using both GC-MS and LC-MS, we are able to offer a comprehensive and robust analysis for those compounds most of which are in the target list of compounds in ASTM test method D8399 and AOAC SMPR 2018.011. This illustrates the proper methods for handling the CRM samples and the appropriate analytical technique used to evaluate every compound. Using both analytical techniques in addition to well-characterized CRMs represents a significant benefit for an accurate and reliable analysis of these harmful chemicals.

P-39

Pesticides Screening Solution: From Data Independent Acquisition to Automatic Targeted Reinjection in Complex Matrix

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The screening and confirmation of pesticides in complex matrix is analyzed using a data independent acquisition (DIA) mode on a high-resolution quadrupole time of flight (Q-TOF) allowing for retroactive analysis of the samples to support the ever-growing list of emerging compounds. Results of targets and suspects are processed using an automated confirmed, questionable, or unidentified designation simplifying the processing of dense Q-TOF data. These identifications are supported by a combination of high-resolution, extended dynamic range, stable accurate mass, and isotopic fidelity with coeluting fragment ions. New software allows for further confirmation of confirmed and questionable analytes through automatic targeted reinjection. In this experiment, broccoli matrix was extracted using QuEChERS sample preparation and spiked with a pesticide mixture of over 200 compounds and 4 heavy internal standards. A calibration curve was produced at 8 concentration levels ranging from 0.625 ng/ml to 100 ng/ml (n = 6) and acquired with an All Ions method. The results showed good mass accuracies, most within a ±2 ppm window and %RSD below 10% for most confirmed compounds at 5 ng/ml. Using the results of the screening data, targeted reinjection was engaged to further analyze compounds listed as confirmed or questionable. In a proof-of-concept experiment this resulted in the unambiguous identification of 18 compounds that were originally designated questionable, of 22 submitted for reinjection. Utilizing Intelligent Reflex targeted MS/MS confirmation workflow, analysts can go from All Ions screening to targeted confirmation without the need to intervene.

P-40

Nontargeted Analysis of Per- and Poly-Fluoroalkyl Substances (PFAS) using LC-QTOF MS and Fluoromatch

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PFAS are diverse synthetic chemicals widely used in industry because of their thermal stability, hydrophobicity, and relative inertness. These “forever chemicals” have been found in food and food packaging and may cause harmful health effects. There are more than 14,000 PFAS compounds known to date (EPA CompTox dashboard) but only a small number of PFAS are screened by targeted PFAS analysis. Non-targeted methods using LC-Q-TOF HRAM MS are

needed for comprehensive PFAS determination. The Agilent 6546 LC-QTOF MS dramatically increases PFAS coverage when acquiring data due to its sensitivity, dynamic range, resolution and mass accuracy stability. To address the bottleneck of processing of non-targeted PFAS data, FluoroMatch is used, a freely available open-source software from innovativeomics.com/software that streamlines PFAS annotation and visualization. FluoroMatch automates file conversion, chromatographic peak picking, blank feature filtering, PFAS annotation based on precursor and fragment masses, and annotation ranking. The software library contains ~7,000 PFAS fragmentation patterns based on rules derived from standards and literature, and the software automates a process to add additional compounds. Optimization of the PFAS analysis workflow will also be discussed.

P-41

Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Food Using a Novel Simplified Sample Preparation Method followed by LC/MS/MS Detection

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Per- and polyfluoroalkyl substances (PFAS) comprise a large group of synthetic chemicals used in industrial and consumer products. The dietary exposure to PFAS substances through foods draws more and more attention, calling for the reliable analytical methods for PFAS determination in food. However, the analysis of PFAS in food matrix can be challenging on sample preparation due to the high variety and complexity of food matrices.
In this study, we developed a method using QuEChERS extraction followed by passthrough cleanup on novel SPE cartridges using LC/MS/MS detection for PFAS analysis in food matrices. Pre-homogenized food sample was extracted using QuEChERS extraction followed with passthrough cleanup on the appropriate SPE cartridges. Food sample size was determined based on the matrix complexity and moisture content. The passthrough cleanup provided fast yet efficient matrix cleanup, and the selection of cleaning cartridges was based on matrix complexity. Sample analysis was performed using an Agilent LC/MS/MS system, with the modified LC system using a PFC-free conversion kit to eliminate any potential interferences or contamination from system. Isotopic internal standards were used for quantitation. The method was evaluated for targets recovery, reproducibility, and matrix effect at 1 ng/mL in food, limits of quantitation (LOQ), and calibration curves range and linearity. The method demonstrated as a simplified and reliable workflow providing acceptable PFAS quantitation results. The sample preparation method simplicity and consistency also make it an easy adoption for adoption in different food matrices, with minimal method modification, save time and labor with the improved lab productivity.

P-42

Analysis of Nitrosamine Impurities in Sartan Drugs using Agilent GC-MS Instrumentation Equipped with the HydroInert Ion Source for Hydrogen Carrier Gas

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Hydrogen as a carrier gas in GC-MS analysis is gaining popularity due the overall lower cost in instrument operation and availability when compared to helium gas. The challenges facing laboratories wanting to utilize hydrogen gas is its reactivity in the EI ion source of the mass spectrometer, which often results in spectral infidelity when compared to the expansive helium-based EI mass spectra library. Agilent has developed and released an extractor EI source, the HydroInert source, designed to improve spectral fidelity with respect to helium by reducing the reactivity of hydrogen in the ion source. A GC-MS equipped with the HydroInert source was used to analyze 9 nitrosamines in sartan drugs to demonstrate how helium carrier gas can be substituted with hydrogen and meet the same criteria as helium-based analysis.

P-44

The use of Short 10 mm Columns for Rapid LC-MS Residue Testing

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The increasing number of samples that are seen in high throughput analytical laboratories such as some environmental testing labs means that fast LC-MS analysis, using triple quadrupole and high resolution (HRMS) instrumentation, is becoming essential. Over the last two decades, many labs have dramatically increased sample throughput using UHPLC, using shorter columns packed with sub-2 micron particles. However, the performance of modern mass spectrometers has continued to evolve. Improved sensitivity and ultra-fast data acquisition capabilities allow further reductions in analytical run times, using specially designed, high throughput columns.
This poster assesses the use of 10 mm columns for the analysis of complex samples, derived from environmental sources. The theoretical considerations of short columns are discussed, starting with van Deemter theory, before moving to a kinetic plot interpretation of their application. The impact of extra-column dispersion on this column format was assessed theoretically and experimentally by systematically varying post-column tubing volume on a Sciex QTRAP® 6500+ LC-MS/MS system and assessing the chromatographic performance of a set of injected analytes. Other considerations, including MS dwell time and LC dwell volume were also assessed.
Finally, a series of applications are presented which demonstrate the benefits that 10 mm length columns can have for high throughput analysis in the environmental sector. The applications will include a method for the determination of 26 illicit drugs (a subset of a larger contaminants of emerging concern assay) and high throughput PFAS analysis.

P-45

PFAS Dark Matter and Slippery Cannabis: Disparate Problems with a Similar Path to a Solution

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PFAS compounds have a long and diverse history of applications yet only recently has sufficient attention been focused on their environmental and toxicological impacts. Despite the tremendous numbers of PFAS compounds in existence, a relatively small number have been fully studied and are commercially tested routinely. The term “PFAS Dark Matter” has emerged to signify the recognized gap between Total Organic Fluorine, Total Oxidizable Precursors, and targeted methods using tandem mass spectrometry. Similarly multifarious, cannabis has a complex chemical composition that includes terpenes, sugars, hydrocarbons, steroids, flavonoids, amino acids, and other compounds of interest. The primary objective of this study was to leverage a relatively novel analytical combination of LC, High Resolution Ion Mobility (HRIM) and QTOF approaches to unravel the complexity seen with existing separation challenges. We used the MOBILion HRIM system based on Structures for Lossless Ion Manipulation (SLIM) to assess cannabinoids and PFAS in a variety of matrices. Accurate mass, isotope spacing, isotope ratios, and mobility aligned fragmentation were used in various combinations for tentative and absolute identification depending upon available standards. In several cases, CCS values were derived providing a unique, molecular identifier that was leveraged to generate 2 dimensional plots of CCS vs. m/z to elucidate trendlines and characteristic subclasses revealing distinctive relationships within and across compound classes. Lastly, previously established CCS values were used to generate reference plots of CCS vs. m/z as a tool to understand potential impact of interferences with known, endogenous compounds where applicable.

P-46

Optimal Separation of Anionic and Cationic Pesticides From Fruits and Vegetables with Unique HPLC Column Selectivity.

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Analysis of polar pesticides presents multiple challenges including adequate retention, separation of critical pairs, and reproducibility. Current HPLC columns available is dedicated to specific mode of analysis, can require long equilibration and hence results in low throughput. In addition, food matrices can add additional challenges due to the presence of complex matrix components including pigments, fats, and sugars that can interfere with the analyte of interest. polar, anionic analytes like Glyphosate will utilize QuEChERS or QuPPE sample preparation techniques, followed by HILIC LC-MS/MS methods for chromatographic retention and separation. Historically, these methods are not user friendly, and lack reproducibility necessary for a commercial laboratory application. In this study, we are presenting a unique HPLC selectivity that provides optimal separation of various anionic pesticides including Glyphosate, Chlorate, Perchlorate, Ethephon, Phosphoric acid-based pesticides, and N-Ac-Glucosamine plus chlorate and perchlorate. In addition, we demonstrate optimal separation and detection of various cationic polar pesticides in both reverse phase and HILIC modes followed by MS detection. Thus both anionic and cationic pesticides can be analyzed in one, reversed phase separation due to the mixed mode capabilities of this unique column. The study demonstrates robust polar pesticide analysis from real sample matrix in addition to providing fast equilibration, optimal retention and peak symmetry for both anionic and cationic pesticides in various food extracts.

P-47

Improved total organic fluorine method for more comprehensive measurement of PFAS in industrial wastewater and river water

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Per- and polyfluoroalkyl substances (PFAS) are high-profile environmental contaminants, many having long persistence in the environment and widespread presence in humans and wildlife. Following phase-out of perfluorooctane sulfonate (PFOS) and perfluorooctanoic acid (PFOA) in North America and restrictions in Europe, PFAS replacements are now widely found in the environment. While liquid chromatography (LC)-mass spectrometry (MS) is typically used for measurement, much of the PFAS is missed. To more comprehensively capture organic fluorine, we developed sensitive and robust methods using activated carbon adsorption, solid phase extraction, and combustion ion chromatography (CIC) to measure total organic fluorine (TOF) in industrial wastewaters, river water, and air. Two extraction techniques, adsorbable organic fluorine (AOF) and extractable organic fluorine (EOF), were optimized and compared using 39 different PFAS, including replacements, such as GenX and perfluorobutanesulfonate. Our AOF method achieves 46–112% and 87% recovery for individual PFAS and PFAS mixtures, respectively, with a 0.3 µg/ L LOD for a 500 mL sample volume. Our EOF method achieves 72–99% and 91% recovery for individual PFAS and PFAS mixtures, respectively, with 0.2 µg/L LOD for a 500 mL sample volume and 0.1 µg/L LOD for 1200 mL. In addition to 39 anionic PFAS, two zwitterionic PFAS and two neutral PFAS were evaluated using the optimized TOF methods. Substantially higher TOF values were measured in industrial wastewater, river water, and air samples compared to LC-MS/MS, demonstrating how TOF methods provided a more comprehensive measurement of the total PFAS present, capturing known *and unknown* organic fluorine.

P-48

Analysis of Contaminants in Cosmetics and Consumer Products by HPLC

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Formaldehyde is widely used in manufacturing of building materials, cars, plywood, polymers, glues and adhesives. It is also commonly used in household items as a preservative and disinfectant. Due to high toxicity and suspected carcinogenicity products are tested to determine free formaldehyde content and ensure the safety of personal care products and other items. Free formaldehyde testing is also important to monitor production of resins and other polymers and to ensure the quality of final materials. Formaldehyde and formaldehyde-releasing agents are commonly used in personal care products, such as cosmetics, as preservatives and disinfectants. Due to high toxicity and suspected carcinogenicity of formaldehyde the products are tested to determine free formaldehyde content and ensure safety of the consumers. HPLC method with post-column derivatization allows for quantifying free formaldehyde in a wide range of products and materials and is the basis of the analytical method listed in Chinese Cosmetic Safety Technical Specification 2015. The method is simple, selective and very sensitive. This method is suitable for the determination of free formaldehyde content in water-based cosmetic products, cream emulsions and gel cosmetics. Potential interferences are either separated from formaldehyde on HPLC column or do not react with post-column reagent and are not detected. The matrices that were used are cosmetics, phenolic resins, fragrances, leather and fabric. Leather and fabric samples were extracted according to ISO 177226-1 and ISO 14184-1. Formaldehyde was analyzed in personal care products as low as 0.0001%. NDELA analysis was as per DIN EN ISO 10130.

P-49

Large Volume Injection of Pesticides Using Low Pressure Gas Chromatography

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Concurrent Solvent Recondensation Large Sample Volume splitless injection (CSR-LVSI, or LVI) is a gas chromatography sample injection technique that overcomes the limitation of using a smaller sample volume of 1 to 2 µL which is typical of conventional splitless injections. Low Pressure Gas Chromatography (LPGC) is a novel technique that has been successfully applied for multi-residue pesticide detection and quantification. The LPGC configuration with the restrictor/guard column facilitates the requirements of a CSR-LVSI and has a potential to improve the sensitivity and lower detection limits. Peak shapes of large volume injections using both acetonitrile and acetonitrile – toluene were evaluated in the range of 5 – 50 µL and the relationship between the peak area and injection volume was established. The calibration parameters were compared for 5, 15 and 25 µL injection in solvent and matrix. The limits of detection and other parameters were compared between the two injection volumes.

P-50

LC-MS/MS Method Development for Ultrashort-Chain and Short-Chain PFAS Analysis in Potable and Non-Potable Waters

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While the current standard tests are focusing on the PFAS with carbon chains of C4 and up using reverse-phased liquid chromatography (RPLC) methodologies, more studies have shown the prevalence and high levels of ultrashort-chain (USC) PFAS (C1-C3) in environmental aquatic systems (e.g., rain, river, ground waters, and wastewaters). USC PFAS are

very polar compounds contributed to at least 40% of total PFAS detected in environmental waters, with trifluoroacetic acid (TFA) being the most abundant and difficult to be analyzed by conventional RPLC. Therefore, a simple and reliable LC method is necessary to fulfill the need for standard analytical workflows of USC PFAS.
This presentation will discuss a proposed ASTM method development (WK80687) for C1 to C4 carboxylic acid and sulfonic acid PFAS analysis in tap waters, bottle waters, and wastewaters. A direct injection method was evaluated by accuracy and precision analysis implementing a unique hybrid HILIC/ion exchange column. A critical challenge was encountered for TFA quantification due to its ubiquitous contamination in reagent solvents used in the lab. A fast LC method with isocratic elution was established for accurate quantification of all C1 to C4 PFAS. The isotopically-labeled C4 compounds, M3PFBS and M4PFBA, were implemented as internal standards to correct various matrix effects in different water samples. This workflow provides convenient setup and high throughput analysis for the lab interested in adding ultrashort-chain compounds to PFAS assay in both potable and non-potable waters.

P-51

The Evolving Landscape of PFAS Detection, an Outline of Methods

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Perfluoroalkyl substances are a group of man-made chemicals widely used in industrial applications and consumer products. Their widespread usage and resistance to degradation has resulted in PFAS being a ubiquitous environmental contaminant and the potential health effects is of growing concern. While many of the long-chain PFAS have been recognized as harmful, alternative compounds have emerged in their place. Short-chain PFAS compounds are considered to be less bio accumulative and toxic than long-chain, but their widespread use has resulted in their increased environmental accumulation. In this work, several methods will be outlined to meet the evolving landscape of PFAS analysis. These methods include EPA methods 1633, 533, 8327, and 537.1 as well as a method for the analysis of ultra-short through short-chain (C1-C4).

P-52

Determination of 3-Acetyldeoxynivalenol (3-ADON) and 15-Acetyldeoxynivalenol (15-ADON) by LC-MS/MS utilizing Differential Mobility Spectrometry (DMS)

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Matrix background and isobaric co-elution can create challenges for routine mycotoxin analysis in LC-MS/MS methodologies. While many can be resolved chromatographically, orthogonal means of separation can assist in the specificity and accuracy of such analyses. Here we demonstrate a simple and effective use of DMS to quantify 3-ADON and 15-ADON within a routine expanded mycotoxin LC-MS/MS methodology.

P-53

Electron activated dissociation (EAD) for pesticide structural elucidation and confirmation in food matrix using the ZenoTOF 7600 system

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The intensive use of pesticides in agriculture has led to the need for rigorous and extensive mass spectrometric analysis in food products to protect humans from potential harm. Non-targeted analysis using liquid chromatography-high resolution accurate mass spectrometry is beneficial for identifying pesticides in food matrices. Identification and structure assignment depend on high-quality MS/MS spectral data. However, traditional fragmentation using collision-induced dissociation (CID) can be too aggressive and results in a limited number of fragments, hence not ideal for diagnostic MS/MS spectra, especially in complex matrices. Electron-activated dissociation (EAD), as an alternative fragmentation method, has shown the potential to produce more robust spectra. The softer approach of EAD produces more diagnostic fragments that can supplement the lack of structural information whenever CID produces non-selective fragments, which can be leveraged for distinguishing coeluting isomers or background interferences. In this work, EAD fragmentation from the ZenoTOF 7600 system was used to perform qualitative pesticide structural identification in several food matrices.

P-54

Measuring PFAS in a variety of food samples: can consumers avoid PFAS with more expensive food options?

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Per- and polyfluoroalkyl substances (PFAS) are known to be in a variety of foods we consume daily. They also pose significant health risks. As the public becomes more aware of these compounds, they might make purchases that they believe decrease the amount of potential exposure to PFAS. This study explored the differences in PFAS concentrations in several food types at different price points.
Eight different food products were selected to represent a wide range of food types, these included ground beef, cake, salmon, tuna, potato chips, seltzer, beer, and ice cream. For each type of food, low-, mid-, and high-priced options were purchased for analysis. Across the different food types, there was no observable pattern correlating cost with total measured PFAS. One notable trend was that, of all the sampled food types, the animal-based proteins tended to have the highest total PFAS overall. Additionally, the animal-based proteins contained the greatest diversity of PFAS compounds detected, with most samples having 3 or more species at quantifiable levels. The only food type to contain no PFAS compounds at any price level was beer.

P-55

Learnings and Challenges Encountered in the Development and Validation of a LCMS/MS Method for the Analysis of PFAS in Drinking Water

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Per- and polyfluoroalkyl substances (PFAS) are commonly found in materials used for their analysis in LCMS/MS, providing a unique challenge for accurate quantitation. While exchanging some LC components or employing the use of a delay column may assist in minimizing the potential for inaccurate PFAS quantitation caused by system related issues, recent analyses have shown other sources involved in the sample preparation can introduce PFAS substances to the water samples through contamination. In this study, both consistent and random contamination of various PFAS substances were found that caused results to fall outside of the EPA specifications. Identification of the PFAS contamination sources encountered and remedial actions taken will be presented. Other general findings regarding the challenges of PFAS analysis will be discussed.

P-56

Sensitive analysis of polychlorinated dibenzo-p-dioxins/furans using gas chromatograph mass spectrometry with cost effective helium saving technology

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Polychlorinated dibenzo-p-dioxins/furans (PCDDs / PCDFs) and their toxicity at trace levels in food and feed are a concern for regulatory agencies. With maximum allowable limits set at sub pg/g levels, sensitivity is of utmost importance for gas chromatography-triple quadrupole mass spectrometry (GC-MS/MS) as a confirmatory method for PCDDs / PCDFs under EU regulations 644/2017 and 771/2017. Helium is typically used as a carrier gas in GC-MS instrumentation due to its high purity, inert nature, and overall performance. However, the dwindling global supply has resulted in shortages and higher costs for laboratories. Hydrogen as an alternative carrier gas presents a possible solution, but the lower viscosity causes reduced pumping efficiency and reduced sensitivity compared to helium, making it difficult to meet regulatory requirements. Therefore, technological solutions are needed to minimize helium consumption without compromising analysis results. In this study, we evaluated the performance of a GC-MS/MS equipped with an advanced electron ionization (AEI) source and Helium saver split/splitless injection module for trace analysis of PCDDs / PCDFs. Quantitation was performed using isotopic dilution and Chromeleon CDS software. The Helium saver split/splitless injection module uses nitrogen for purge and split line flows to reduce helium consumption. Compliance with the EU regulations for analysis of dioxins in food was achieved. Ion ratios and relative response factors were within ±15% and ±30% respectively, with precise quantitation at fg levels and stable injection repeatability over long injection sequences. Helium consumption was reduced 8-fold with no reduction in sensitivity and without the need for method re-optimization.

P-57

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Helium carrier gas for GC has been the standard choice due to its chromatographic efficiency and inertness, particularly important with mass spectrometry analysis. However, due to pricing and availability, laboratories are considering other carrier gases for GC-MS analyses. Hydrogen is a common alternative to helium and offers several benefits such as increased linear velocity resulting in shorter run times, and the use of hydrogen generators to create gas on-demand providing a sustainable and cost-effective source of carrier gas. However, there are challenges when using hydrogen with GC-MS analysis. In addition to the flammability risk, hydrogen has effects in the MS ion source and ionization process, often resulting in a decrease in sensitivity or atypical creation of molecular fragments. Laboratories using GC-MS for the analysis of pesticides may benefit using hydrogen carrier gas, but this may require method re-optimization and validation. In this study, the TSQ 9610 GC-MS/MS with an Advanced Electron Ionization (AEI) source is evaluated for pesticide analysis using hydrogen. This solution is capable of identifying and quantifying pesticides at 0.01 mg/kg concentration level with hydrogen carrier gas. The proposed method provides a viable alternative for laboratories performing the testing for pesticide residues in a range of food matrices and looking at ways to circumvent helium price increases or supply shortages. While hydrogen has a negative impact on detection sensitivity, the use of the AEI ion source together with a few changes in the choice of transitions can offset these and allow comparable performance at reduced operating costs.

P-58

Reliable Analysis of Ethylene Oxide and 2-chloroethanol in food samples using Gas Chromatography Mass spectrometry

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Ethylene oxide (EO) has been used illegally for the preservation of a wide variety of dry food products including seeds, cereals, spices, herbs, locust bean gums, etc. Since residues of EO are considered potentially mutagenic and carcinogenic, thus EO and its derivatives in food need to be monitored closely.
EO is analytically challenging as it is a small molecule and highly volatile with a boiling point of only 10.7 °C. During sample preparation special precautions must be taken to avoid analyte losses through evaporation. The molecule is poorly retained on capillary GC columns and the most abundant transition cannot be employed if nitrogen is used as a collision gas. Furthermore, EO can be converted into 2-chloroethanol (2CE), 2-bromoethanol and ethylene glycol via chemical reaction with the sample. The residue definition of EO according to Reg. (EU) 2015/868 includes two compounds - ethylene oxide and 2-chloroethanol (2CE), reported as the sum of EO and 2CE expressed as EO. The EU Maximum Residue Levels range from 0.02 to 0.1 mg/kg depending on the commodity.
This study shows how both EO and 2CE can be analysed with high sensitivity and selectivity using a triple quadrupole GC-MS system with argon as the collision gas. The developed method provided excellent linearity, quantitation and stability of response. The results for 2CE in a variety of matrices agreed with results obtained by an independent ISO 17025 accredited laboratory. Further details will be presented in the poster to enable the method to be implemented in any laboratory.

P-59

Extended automation for on-line or off-line micro-SPE clean-up of QuEChERS extracts for GC-MS/MS analysis of pesticides residues in food

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As demand for routine pesticides residue testing increases, laboratories are under pressure to analyze many different food commodities for hundreds of different pesticides in compliance with statutory Maximum Residue Levels (MRLs). However, the process is often slowed by the need for manual sample extraction and clean-up before chromatographic separation. A quicker, simpler, more generic method with minimum clean-up may facilitate higher sample throughput, increased method scope and lower cost per sample, but it might also lead to extracts with high concentrations of matrix co-extractives which would contaminate the analytical system more quickly. A more effective clean-up procedure that can remove more of the co-extractives without impacting on the recovery of pesticides of interest or extending the overall analysis time, is required.
This poster details the use of a more efficient automated solid phase extraction using an optimized blend of sorbents contained in miniaturized cartridges (µSPE). This system is available as a dedicated tool for the Thermo Scientific™ TriPlus™ RSH robotic autosampler, which supports on-line or off-line automated workflows, for GC-MS/MS and LC-MS/MS. Sample handling automation is essential for laboratories that want to extend unattended operations, for reducing labour time and increasing efficiency and productivity. To enhance this benefit, an additional automated step in the overall workflow was developed to include the automatic preparation of the calibration standards and automatic sample dilutions. This is further reducing manual operations and exposure to human errors, resulting in higher overall sample throughput with better data quality. More details will be presented in the poster.

P-60

An LC-Orbitrap-MS based non-targeted metabolomics approach for investigation of honey adulteration

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Thermo Fisher Scientific, India Customer Solutions Center, Ghaziabad, India[1] and San Jose CA[2]

Honey, a sweet, flavorful, and nutritious food produced by honeybees is widely consumed by humans as it has several therapeutic effects due to its antioxidant, antimicrobial, and anti-inflammatory properties. Also, honey has been used in the treatment of wounds, burns, and gastric ulcers. The high price, low production, and complex nature of honey have attracted more attention towards adulteration, which adversely affects consumer health. Common honey adulterants are sugar syrups, i.e. corn syrup, high-fructose corn syrup, inverted syrup, and rice syrup that is added to replace pure honey. Hence, there is a critical need to develop a method to identify and differentiate the adulterated honey to control honey quality and safety. Detection markers of honey adulteration include polysaccharides, 5 di-fructose anhydrides (DFAs), as well as 7- and 2-acetyl furan-3-glucopyranoside (AFGP). There is currently no available method that can simultaneously detect all the common sugar syrups present in adulterated honey samples. Therefore, there is a need to develop a simple, rapid, and sensitive detection method that simultaneously detects sugar syrups. To identify oligosaccharides, polysaccharides, and phytochemicals, a unique featured accurate and sensitive mass spectrometer for data acquisition and data processing is required, followed by retrospective data mining.
We show an optimized method for qualitative screening of honey by using simple dilute-and-shoot followed by acquisition in combination with UHPLC and the Thermo Scientific™ Exploris Orbitrap™ 240 high-resolution mass spectrometer. The chromatography and mass spectrometry data processing criteria were set as per the SANTE/11312/2021 guidelines.

P-61

A novel cloud-hosted spectral library application used in the development of a Per- and polyfluoroalkyl substances (PFAS) method in pork meat using an LC-Orbitrap high-resolution mass spectrometer

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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 12000 known PFAS (with more PFAS being actively discovered) and only a limited number of certified reference standards commercially available for routine targeted analysis. This work describes a targeted LC-HRAM method with excellent sensitivity and specificity demonstrated in pork meat. A cloud-hosted spectral library with built-in curation tools was used to build a library of highly curated spectra used in the confirmation process. Advantages and features of this cloud-hosted application will also be discussed.

P-62

Microplastics Analysis using Accelerated Solvent Extraction (ASE) and Pyrolysis Gas Chromatography / Mass Spectrometry (Pyr-GC/MS).

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Micro- and nano- sized plastics (MNPs) are considered contaminants of emerging concern for both environmental and human health due to their ubiquity and small size. Assessing microplastic exposure levels in humans is important to understand.
Rice is a staple food for more than half of the global population and it poses the highest food yield among the cereals providing >20% of the human dietary energy worldwide. In certain countries (e.g. Bangladesh and Cambodia) rice can constitute up to 70% of the dietary energy intake. Most of the rice available for consumers is packaged in plastic, but it is not known if packaging could be a contributing source of plastics. Rice is also sold pre-cooked, only requiring the consumer to microwave the rice inside the plastic packaging it is sold in. As a convenience food, it passes through several additional stages of industrial processing that could potentially lead to additional plastic contamination. Standardized mass concentration-based methods for the quantification of plastics in food are currently unavailable.
This work investigates plastic contamination in consumer rice products using a novel technique for plastics extraction and analysis in food. Rice samples were analyzed using a quantitative method consisting of Accelerated Solvent Extraction (ASE) followed by double-shot pyrolysis gas chromatography/mass spectrometry (Pyr-GC/MS) to estimate the mass concentration of selected plastic polymers in rice. The advantage of this methodology is that it is particle size independent and provides concentrations as a total mass of plastic per sample.

P-63

Utilizing a modern Chromatography Data System to power productivity in quantitative pesticide residues analysis

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The analytical requirements for the quantitative analysis of hundreds of different pesticide residues at low levels in a wide variety of samples are complex. With the diversity of the chemical properties of an ever-growing list of target compounds, laboratories increasingly need to utilize LC-, GC-, IC- MS/MS and HRAM technology to enable comprehensive testing. Furthermore, setting-up the acquisition methods and data processing methods for large volumes of raw data, and then ensuring results are compliant with the SANTE analytical quality control criteria is not only time consuming, but can be a source of errors. The Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) version 7.3.2 is a fast, intuitive, user friendly data system, which can control IC, GC, and LC -MS instruments and reduce training requirements and time in the laboratory. A new unique feature is dual sequence data comparison which merges sequences from two different instrument (e.g. LC-MS and GC-MS) so the data processing of pesticides common to both techniques can be undertaken simultaneously in a single view, providing orthogonal cross confirmation of identity and increased confidence in results. This is especially the case for complex samples where interferences are more prevalent. 'Dynamic data processing' and 'linking' ensures selections and changes are instantly reflected in the data and results, saving valuable data processing time. The poster will provide more in-depth information on these and other automation tools such as filtering and flagging options via conditional formatting, 'SmartLink', customizable view settings, and report templates to improve productivity in the pesticide's laboratory.

P-64

Identification of microplastics in water and food using pyrolysis GC with high resolution Orbitrap mass spectrometry

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Microplastics are small particles made from synthetic polymers with a diameter typically ranging between 5 mm and 1 µm, while nanoparticles cover particles sizes of sub 1 µm. Because of their small size they can migrate very quickly and can be easily incorporated into the food chain. Microplastics may not only consist ofthe pure synthetic polymer, but also include residuals of the monomer, plasticizers, flame retardants, and other toxic additives that can have a negative impact on human health.
Samples of storm water, milk and meat were analyzed using a pyrolizer (Frontier Laboratories) mounted on a Thermo Scientific™ Orbitrap Exploris™ GC 240 mass spectrometer. The mass spectrometer was operated in full-MS mode using 60,000 mass resolving power.
Storm water was spiked with deuterated polystyrene (D5-PS) and then filtered to collect particulates prior to determination. Milk and steak samples were freeze dried, milled, spiked with D5-PS and then extracted by pressurized liquid extraction prior to analysis
A series of polymer standards were subjected to pyrolysis to find characteristic fragmentation products were used for polymer identification in the resulting pyrograms To simplify further data treatment during the analysis of samples, a targeted processing method was also created. Benzene, naphthalene, and fluorene were found in the stormwater while styrene, allylbenzene, α-methylstyrene, and toluene were identified in the samples of milk and steak.
The study demonstrates Py-GC-Orbitrap is a robust tool for the confirming the presence and identity of microplastics in different sample types. The presenation will provided further details on the overall method.

P-66

Analysis of Pesticide Residues in Cannabis using QuEChERS Extraction, ChloroFiltr® dSPE Cleanup and LC-MS/MS

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With the recent trends in legalization, interest in cannabis and cannabis-based products (e.g. concentrated oils, soda, candy and other edible forms) have dramatically increased. Pesticide residues can pose significant health risks, especially with chronic exposure. This Poster outlines a QuEChERS method for the simultaneous analysis of cannabis for 64 pesticides. Most of the LC-MS/MS amenable compounds on the Californnia monitoring lists of pesticides. Sample purification is carried out using UCT's new cleanup product SpinFiltr™, which combines the convenience of classical dispersive-SPE (dSPE) with an ultrafiltration tube containing a 0.2 µm filter membrane to simultaneously remove unwanted matrix components and filter the sample prior to LC or GC analysis. The SpinFiltr™ dSPE tube uses PSA, C18 and ChloroFiltr® sorbents for sample cleanup. ChloroFiltr® is a unique polymeric sorbent designed for the removal of chlorophyll and unlike graphitized carbon black (GCB), does not result in the loss of planar analytes. Liquid chromatography, using a SelectraCore® C18 column, coupled to tandem mass spectrometry (LC-MS/MS) is used for analysis of the pesticides.
The method was evaluated by fortifying cannabis samples with each compound at varying concentrations (n=5 each). The average recovery obtained was predominantly in the range of 70-100% and the reproducibility ≤10%. With the widespread legalization of cannabis, this simple method will be beneficial for any research facility wanting to implement regulatory testing.

P-67

Investigations into Pesticide Charge State Site Isomers with Ion Mobility and High Resolution Mass Spectrometry

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The testing for pesticides in food and other commodities is an important step in guaranteeing consumer safety. Typical analytical methodology for pesticide testing includes liquid chromatography-mass spectrometry (LC-MS) to detect ions based on retention time, mass/charge ratios, and the relative abundance of characteristic product ions (ion ratios). In particular, the ion ratios can be variable and has led to wide tolerances in regulatory methods. One source of this variability is the formation of different charge state isomers (protomers, sodimer, and potassimers) that yield different product ions and can be influenced by matrix, solution, and ion source conditions. In this study, we explored the use of ion mobility with high resolution mass spectrometry to gain a greater understanding of the charge state isomers and their influence on observed ion ratios. Ion mobility spectrometry (IMS) is a fast, gas phase separation technique that can be readily incorporated into an LC-IM-MS arrangement to add an extra dimension of separation and characterization. Using the IMS dimension, different charge-site isomers can be resolved based on their shape and the difference in their collisional cross section (CCS). LC-IM-MS was used to generate a pesticide CCS database and screen for residues in food commodities. For some residues, separation of different charge site isomers required the use of a high resolution cyclic IM device. This device was used to resolve and determine CCS values for isomer species of indoxacarb, spinosad, fenpyroximate, epoxiconazole, metaflumizone, and avermectin.

P-68

Pushing PFAS Possibilities: The Hunt for Ultra Sensitivity to Reach ppq EPA Health Advisory Levels

Kari Organtini, Gordon Fujimoto, Henry Foddy, Nicola Dreolin, Stuart Adams, Ken Rosnack, Naren Meruva, Peter Hancock

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PFAS are well known chemicals with a variety of commercial and consumer uses, characterized by their extreme stability and structural diversity. PFAS are highly persistent and toxic contaminants that accumulate in humans, animals, and the environment. Detection requirements for PFAS have been getting more challenging as advisory and regulatory limits continue to be updated. In June 2022, the US EPA tightened its health advisory levels (HALs) in drinking water from 70 ng/L to 0.004 ng/L for PFOA and 0.02 ng/L for PFOS. Additionally, final HALs were set for PFBS at 2000 ng/L and HFPO-DA (commonly referred to as GenX) at 10 ng/L. The near zero PFOA and PFOS levels pose analytical challenges regarding instrument sensitivity and sample preparation conditions to limit contamination. The goal of this work was to show with proper care during sample preparation and analysis using a highly sensitive mass spectrometer, these near zero limits are possible to detect. All samples were prepared in a typical laboratory environment, taking extreme care to minimize PFAS contamination. This poster will discuss best practices for sample preparation to reduce contamination, discuss LOQs for each compound and method recovery values will be shared.

P-69

Development and Performance Evaluation of a Method for the Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Foods of Animal Origin

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Cases of PFAS contamination of foods have become more prominent in the media, causing a steep rise in concerns

about potential health implications. To better understand dietary exposure and health risk, 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. Increased sample preparation complexity is 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. Further, an interlaboratory study was performed to evaluate method performance for PFHxS, PFOS, PFOA, and PFNA quantitation in fish. Overall, detection and quantitation limits were determined to be in the sub-ng/g range for all matrices. Recoveries were within FDA criteria with utilization of isotope dilution for accurate correction of recovery during calculation of PFAS concentration in samples. Each of the seven participating labs successfully implemented the method and demonstrated acceptable accuracy, trueness, repeatability, and reproducibility. These studies confirm that the described method is suitable for compliance testing in accordance with EU regulations and for risk assessment purposes.

P-70

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

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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 System 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 in order 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-71

A smart clean-up approach to reduce matrix effects and isobaric interference for multi-residue pesticide analysis, based upon LC-MS/MS, of the difficult matrices, black tea and cocoa beans

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Applying a generic QuEChERS protocol without clean-up is challenging when faced with complex commodities due to the abundance of endogenous components, such as fats, phospholipids, pigments, and other phytochemicals. These components are known to cause matrix effects and isobaric interference, which negatively impact detection and quantitation of pesticide residues. Adding a clean-up step after extraction helps to remove matrix components to obtain reliable results, improving sensitivity and selectivity, and maintaining instrument robustness. Using Solid-Phase Extraction (SPE) with a pass-through protocol allows pesticides to pass through the stationary phase, whereas the matrix components are retained on the SPE sorbent material.

The objective of this work was to establish the performance of a method based upon QuEChERS but using a simple pass-through SPE cleanup with Oasis™ PRiME HLB catridge prior to UPLC-MS/MS using ACQUITY™ I-Class UPLC™ System coupled with Xevo™ TQ-XS Mass Spectrometer. We optimized a two-step protocol whereby the QuEChERS extract was pushed through the cartridge, the first milliliter discarded, and the remaining eluate collected for LC-MS/MS. The performance of the method has been successfully evaluated at 0.01 and 0.1 mg/kg in tea and cocoa, with most of the 395 analytes exhibiting recovery and repeatability within the tolerances set in the SANTE guidelines: 70-120% and ≤20%, respectively. A simple pass-through SPE protocol with Oasis PRiME HLB cartridge was proven to be a quick but effective alternative to dilute and shoot or dispersive SPE and has been shown to be suitable for checking MRL compliance for pesticide residues in tea and cocoa.

P-72

Development and validation of a method for the determination of aminoglycosides in foods using LC-MS/MS with a zwitterionic HILIC stationary phase

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Aminoglycosides (AMGs) are broad-spectrum antibiotics that have bactericidal activity against aerobic bacterial infection and are commonly used as veterinary drugs on food-producing animals and in human medicine. Thus, it is important to monitor residues in food to control AMG use. Many countries have established maximum residue limits (MRL) for aminoglycosides approved for use on animals. AMGs are often analyzed in honey, eggs, milk, tissues, and biofluids of food-producing animals for control and monitoring purposes. AMGs are highly polar compounds and show little to no retention in reversed phase columns. Although ion-pairing reagents have been utilized successfully to chromatograph AMGs on C18 columns, when used with liquid chromatography tandem mass spectrometry (LC-MS/MS) this approach suffers from ion suppression and contamination of the LC and MS/MS systems. The introduction of hydrophilic interaction chromatography (HILIC) provided a more MS-compatible option for the analysis of polar compounds. Here we show the results from the successful evaluation of the Atlantis™ Premier BEH™ Z-HILIC column, which has a sulfobetaine zwitterionic chemistry, for the determination of AMGs. Samples of milk, eggs, and honey were extracted using a solution that contained 10 mM ammonium acetate, 0.4mM ethylenediamine tetraacetic acid (EDTA), 0.5% NaCl, and 2% trichloroacetic acid (TCA) and subjected to clean-up by Solid-Phase Extraction (SPE) on Oasis™ HLB cartridges prior to LC-MS/MS. The method was successfully validated according to Commission Implementing Regulation (EU) 2021/808 and is suitable for reliable determination of residues to check compliance with MRLs and in cases where use of the substances is not allowed.

P-73

Comparative Analysis of Nitrogen Carrier to Helium for APCI Gas Chromatography in Food Matrices

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Gas chromatography-mass spectrometry/mass spectrometry (GC-MS/MS) is a powerful analytical technique used for the detection and quantification of pesticide residues in food and environmental samples, traditionally using helium as a carrier gas. There has been difficulty sourcing helium worldwide for well over a decade, leading to significant rises in price and creating the demand for alternative carrier gases for gas chromatography. Nitrogen is readily available, relatively cheap, inert and safe compared to other options such as hydrogen. This work demonstrates the transfer of GC-APCI MS/MS methods run with helium carrier gas to nitrogen using an atmospheric pressure gas chromatography (APGC™) ionization source. APGC demonstrates improvements in selectivity and sensitivity when compared to electron ionization (EI) analysis due to the generation of abundant molecular ions. Furthermore, APGC achieves consistent chromatographic performance when using helium or nitrogen carrier gas.
A routine method of >200 pesticides was run on a Xevo™ TQ-S micro System using both carrier gases. A cucumber extract

was prepared using a QuEChERS based workflow. Extracts of infant food (cottage pie) were also prepared for pesticide analysis using nitrogen as the carrier to show the equivalent performance of each gas when using APGC. Switching between carrier gases while maintaining separation efficiency is achieved using a smaller diameter column and lower carrier gas flowrate for nitrogen.

P-74

Determination of acidic herbicides in water using liquid chromatography-tandem quadrupole mass spectrometry with direct injection

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Herbicides are a specific group of plant protection products used to treat a variety of weeds such as crop rotation, tillage, and fallow systems. Acidic herbicides comprise families of compounds that include derivatives of benzoic acid (e.g. dicamba), acetic acid (e.g. 2,4-dichlorophenoxyacetic acid [2,4-D]), and other miscellaneous acids. Herbicides can enter surface water bodies either directly through spray or indirectly via surface water runoff. Herbicides can also reach water-bearing aquifers below ground (groundwater) from applications onto crop fields and seepage of contaminated surface water, Surface and ground water is pumped, piped, or diverted for use in the supply of drinking water.
The presence of pesticides in drinking water is monitored globally. The EU Drinking Water Directive sets a maximum limit of 0.1 µg/L for individual pesticide residues present in a sample (0.5 µg/L for total pesticides). In the USA, drinking water is regulated under the Safe Drinking Water Act, where there are varying maximum contaminant levels for each residue. There is a need for reliable analytical methods for monitoring acidic herbicides in drinking water. Historically, methods have been based upon using liquid-liquid extraction or solid-phase extraction (SPE) as a concentration step. This poster will demonstrate a rapid method for the determination of a range of acidic herbicides in water samples using large volume direct injection on ACQUITY™ Premier HSS T3 column coupled with Waters™ ACQUITY Premier UPLC™ I-Class System and the Xevo™ TQ Absolute Mass Spectrometer to achieve best degree of performance and ultra-high sensitivity quantitative method. The calibration curves were run from 0.01 to 0.2 ug/L in drinking water. Data were acquired and processed using waters_connect™ software.

P-75

A Simple, easy, and efficient way to process and review data for large methods using the MS Quan app within the new waters_connect™ software

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Running a targeted method for quantitation of hundreds of analytes, across many batches of different samples, presents food testing laboratories with several challenges. Issues associated with developing, managing, and curating error-free acquisition and data processing methods and the review of data can be both time-consuming and cumbersome. To help overcome these daily challenges, we are showcasing the new MS Quan app that is included within the new waters_connect™ software platform.

We will demonstrate the usability of the software by generating a processing method from acquired data, efficiently process, and review the quantitative data with a focus on a workflow utilizing exception focused review.

We will demonstrate the easy of setting up batch QC criteria such as calibration correlation, residuals, QC check, blanks, internal standard response, ion ratio and retention time tolerances, as per documented guidelines or bespoke values. A batch dashboard immediately gives the user an overview of the health of the batch and highlight potential areas that require user intervention to aid laboratory efficiency. There are many enhancements that allow users to view each chromatogram, but this is also made easier with the ability to view many chromatograms on one screen at once, totals, isotope dilution, standard addition and many more new features are available now. Come and join use for a glimpse of this new user-friendly software.
waters_connect is a trademark of Waters Technologies Corporation.

P-76

A Quantitative Lateral Flow Immunoassay for Measuring Glyphosate

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Glyphosate (trade name Roundup™ herbicide) is one of the most used herbicides that control broadleaf weeds and grasses. The widespread use of glyphosate makes it ubiquitous in the environment. Researchers have found its presence in our food, soil, groundwater and surface waters. Recent studies have raised health concerns on glyphosate exposure even though the toxicity of glyphosate is still under debate. The international agency for research on Cancer (IARC) classified glyphosate as a probable carcinogen in 2015. The instrumental method for analysis of glyphosate is available including HPLC and LC-MS/MS. However, the sample preparation and testing procedure are relatively complicated and time-consuming. In this study, a simple quantitative rapid test (Glyphosate-V™) for measuring glyphosate was developed and evaluated with applications on water, wheat and oats. The samples were extracted by distilled water, filtered, derivatized and mixed with a diluent, then directly applied to the test strip device. The results generated from Glyphosate-V test showed a very high degree of linearity (r2 = 0.999), with the test ranging from 2 to 1000 ppb for water, and 25 to 3000 ppb for wheat and oats. The limit of detection (LOD) is 2 ppb for water and 25 ppb for wheat and oats. Comparative study indicates that the results generated from Glyphosate-V™ testing correlated well with the results of LC-MS/MS. The time required from sample preparation to result is less than 20 minutes. In conclusion, the Glyphosate-V test can be used as a screening tool for glyphosate detection in food and environmental samples.

NACRW Veterinary Drug Residue Working Group Abstracts

Comparison of the Low versus High Resolution-based Confirmation Criteria for Veterinary Drug Residues in Food Control

Anton Kaufmann, Official food control authority of the Canton of Zurich, Zürich, Switzerland

Tandem quadrupole based analyte confirmation criteria are well defined and widely accepted. This is not yet the case for high resolution mass spectrometry (HRMS) based measurements. Using the unit mass resolving quadrupole of a Q-HRMS instrument and monitoring two HRMS resolved product ions clearly outperforms any conventional tandem quadrupole based multi reaction monitoring (MRM) confirmation. Hence there is the open question, if reliable HRMS based confirmation is achievable by using wider mass isolation windows (e.g. SWATH) or if the monitoring of one product ion is acceptable.

Progress of the Veterinary Drug Residue Collaborative Study, 2022-2023 (Round 1)

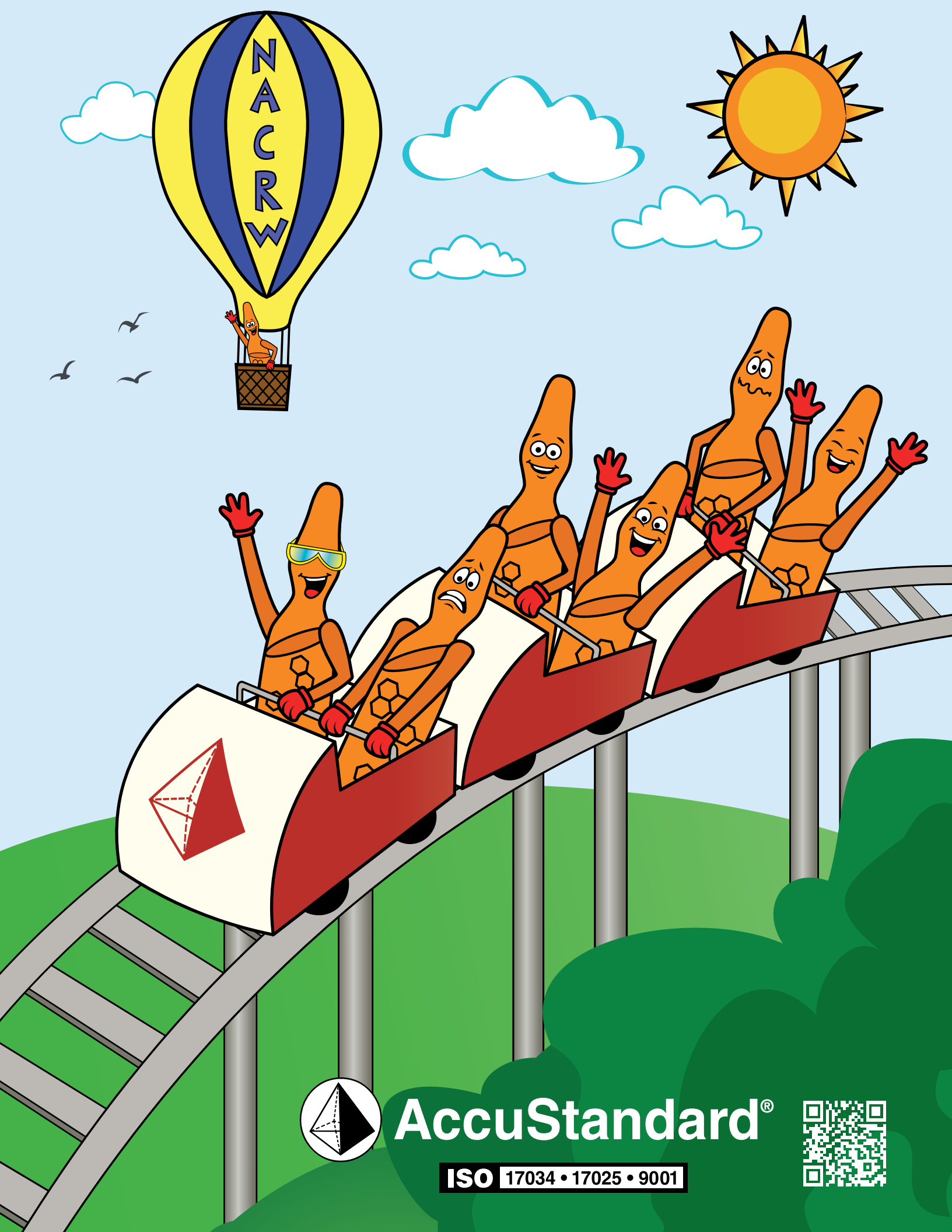
Maïwenn Le Floch and Eric Verdon, ANSES, the French Agency for Food, Environmental and Occupational Health & Safety, Laboratory of Fougeres; France

In 2019, the Working Group started to talk about the organization of an inter-laboratory collaborative study to evaluate the screening practices for veterinary drug residues carried out using various new generation mass spectrometry instruments. Three “Rounds” were planned, each of them focusing on two different commodities. Round 1 organization started in the fall of 2021 led by the reference laboratory of Anses-Fougères in France. This laboratory have sent to participants in summer 2022 multiple trays of vials consisting of spiked final matrix extracts and relevant calibration standards. All the data have been collected in autumn 2022, and since have been processed. During this meeting will be presented the major outcomes of the study.

Development of HRMS Food Residue Megamethods

Steve Lehotay, USDA ARS, Wyndmoor PA, USA; Jian Wang Canadian Food Inspection Agency, Calgary, CANADA; Jon Won, USFDA Center for Food Safety and Applied Nutrition, College Park, MD, USA; Sherri Turnipseed, USFDA Animal Drugs Research Center, Denver, CO, USA

A panel of scientists will update progress in their laboratories on the development, validation and implementation of analytical methods for a large number of chemical contaminants using LC- HRMS. Aspects of extraction techniques, instrumental optimization, and data evaluation will be discussed with application to both pesticide and veterinary drug analysis.



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