Online NACRW

NORTH AMERICAN CHEMICAL RESIDUE WORKSHOP

July 26-30, 2021

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FUTURE MEETING DATES

2022
July 26-29
Marriott Harbor Beach Resort
Fort Lauderdale, Florida

2023
July 25-28
Marriott Harbor Beach Resort
Fort Lauderdale, Florida

2024
July 24-27
Marriott Harbor Beach Resort
Fort Lauderdale, Florida

Dear Attendees, Exhibitors, Sponsors and Guests:

Welcome to the 57th, North American Chemical Residue Workshop! We extend a warm greeting to our long-time attendees, our international guests, and our first time participants. We would like to especially thank our Exhibitors and Sponsors for their generous support. Their financial contributions have made it possible for outstanding presentations, while maintaining affordable registration fees for attendees. The fantastic technical sessions, interactive poster presentations, and relaxed atmosphere have made NACRW a favorite event for many! While we can't meet in-person this year, we are looking forward to the many presentations and opportunities to interact virtually.

We hope you will make plans to attend the NACRW Reference Materials and Veterinary Drugs Working Groups on Monday. The Reference Materials Working Group will present the 1st Edition of "Reference Material Use in Trace Analysis" which will be followed by Open Forum discussion. The Veterinary Drugs Working Group will consist of presentations and Open Forum discussion on optimal MS-based analyte identification criteria. In addition, a collaborative study of multiple matrices and analytes is being planned to evaluate analyte identification criteria to minimize the rate of false positives and false negatives.

Since 2015, NACRW has presented the Excellence Award to Scientists based on scientific achievements, highly influential publications, innovations, and lifetime contributions. This year's award topic is Excellence in Detection Techniques. We are pleased to announce the recipient of the 2021 NACRW Excellence Award is Dr. Alexander Makarov. Dr. Makarov is recognized for his pioneering efforts in the commercial development of the Orbitrap Mass Spectrometer, resulting in the worldwide advancement of high-resolution mass spectrometry from chemical residue determinations.

Our Program Committee has developed a fantastic technical program for you this year. It includes a variety of chemical residue related subjects and special interest topics which include: Sample Preparation, Advancements in QA/QC, Cannabis, Single & Multiresidue Pesticides Methods, Perfluorinated Compounds Environmental Contaminants, and New and Advanced Technology in Residue Analysis. In addition to these topics, there will be the Pesticide Residue Forum which allows for attendees to ask questions to our panel of experts. If you have a question, please submit your question at registration or through the chat feature in the virtual platform.

In addition to our oral sessions, please plan to attend the poster sessions, exhibitors, and vendor technical presentations. The poster authors will be presenting their posters at designated times listed in the program. This is a great opportunity to engage the authors and ask questions. NACRW offers student poster awards, sponsored by FLAG Works, Inc. and the ACS Journal of Agricultural and Food Chemistry. The students will be attending the workshop and giving short presentations on their posters during Session 1 of the conference. The students will also be available at their virtual posters during designated times to have conversations and answer questions. During the workshop, we encourage attendees to visit our exhibitors to learn more about the products and services they offer for all your testing needs. In the EventsAir virtual platform, you have the option of requesting live chats, posting questions through the chat feature, and read exhibitor provided literature. We are pleased to offer Technology Presentations between sessions during the week. This is a great opportunity to hear about the latest developments and exciting new products or applications.

I would like to thank the fantastic volunteers who have helped make this virtual event a reality. To the 2021 NACRW Organizing Committee, Program Committee, especially Katie Carlos, Michael Filigenzi, and Executive Director, Teri Besse; it has been a pleasure to work with you, and I extend my heartfelt appreciation for all their time and commitment to the workshop. I also want to thank NACRW for this opportunity, it has been a rewarding experience.

We hope you enjoy your time at NACRW!

Sincerely,

Alexandria Bush, 2021 Organizing Committee President
Katie Carlos and Michael Filigenzi, 2021 Program Committee Co-Chairs
2021 Organizing Committee and Program Committee Members

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Vendor Technology Presentations

Monday, July 26, 2021
10:15-10:45 am
RESTEK Corporation

New Technology Solutions for Pesticides Analysis

Joe Konschnik, RESTEK Corporation, Bellefonte, PA, USA

RESTEK will present two new technologies for the sample preparation and analysis of pesticides in foods. The first will describe a new-to-world patent pending hybrid HPLC Stationary Phase for the analysis of underivatized polar pesticides. The second will describe a semi-automated workflow for the extraction, clean-up and analysis of pesticides.

Analytical challenges with the analysis of polar compounds motivated the development of a hybrid ion exchange/HILIC column that offers the balanced retention and separation of ionic and other highly polar compounds. With the column, LC-MS/MS workflows were developed for the direct analysis of glyphosate, aminomethylphosphonic acid (AMPA), glufosinate, as well as other 14 highly polar contaminants listed in the Quick Polar Pesticides Extraction (QuPPe) procedure in a spinach matrix. Ultrashort chain PFAs will also be shown.

In-line sample prep (ILSP) provides a semi-automated cleanup procedure for the analysis of pesticide residues in food by LC-MS/MS. ILSP selectively retains matrix components from the sample extract and can be utilized as a standalone workflow or integrated into an existing QuEChERS workflow. In these experiments, ILSP was applied to multiple challenging commodities representing a wide range of compositions including soy meal, avocado, whole orange, black tea, and hibiscus tea for the analysis of 61 pesticides. This solution provides a novel, semi-automated approach to reduce the abundance of matrix components entering the analytical column and MS source resulting in a decrease in instrument contamination and an improvement in data quality.

Monday, July 26, 2021
12:40-1:10 pm
SCIEX

Highly sensitive quantification and selective identification of pesticides in food with Zeno MRM^{HR}

Robert A. Di Lorenzo[1], Lukasz Rajski[2], Jianru Stahl-Zeng[3], Jason Causon[1]

[1]SCIEX, Canada; [2]EURL-FV, Universidad de Almeria, Spain; [3]SCIEX, Germany

In order to ensure safety in the global food supply, testing for adherence to federal and international requirements is necessary. The tests monitor for chemical residues, including pesticides, microbial and fungal toxins, and microbiological hazards. Traditionally, pesticide residue analysis has been performed by triple quadrupole mass spectrometers, due to their sensitivity and quantitative power. Accurate mass instruments can afford additional levels of confirmation, however they have traditionally suffered from a lack of sensitivity and precision, especially when performing MS/MS experiments to meet the testing requirements for the regulatory guidelines.

The technological advancements in the ZenoTOF 7600 system combine qualitative flexibility and quantitative power for the most demanding sample types and workflows. Previously, QTOF mass spectrometers have suffered from duty cycle losses as a result of mating time-of-flight (TOF) analysis, a pulsed measurement technique, with the continuous beam coming from the quadrupole ion path. A series of ion-staging events and potential energy sequential ion release, with high-capacity ion traps, allow for duty cycle losses to be mitigated and for MS/MS sensitivity gains of 4-25x. The cell also has the ability to perform both collision induced dissociation (CID) and electron activated dissociation (EAD) experiments for high-resolution MS/MS flexibility.

The developed quantitative method on the ZenoTOF 7600 system was applied to a variety of food matrices in order to determine LLOQs, and compare the relative pesticide burden between produce that was traditionally farmed and produce purchased from an organic grocer.

Tuesday, July 27, 2021
12:05-12:35 pm
ThermoFisher Scientific

PFAS in NC: Analysis by Isotope Dilution Solid Phase Extraction and LC-MS/MS

Allen Martin, North Carolina Department of Environmental Quality, Raleigh, NC, 27607, USA

Per- and polyfluoroalkyl substances (PFAS) have been commercially produced since the 1940s. The unique chemical properties of these compounds resulted in their use in a multitude of applications. This long-time use, coupled with their chemical stability, has led to their ubiquitous presence in the environment. North Carolina specifically is considered the state with the third-highest level of PFAS exposure. In 2017, investigations were conducted regarding the presence of GenX in the Cape Fear River basin. In 2019, the PFAS testing laboratory was established within the NCDEQ water resources section to monitor PFAS levels throughout the state. Currently, we employ a method of isotope dilution solid phase extraction and analysis by LC-MS/MS for all our PFAS analysis.

Wednesday, July 28, 2021
12:15-12:45 pm
Agilent

Automated MRM Method Development for Pesticides Using the Agilent MassHunter Optimizer for GC/TQ

Anastasia A. Andrianova, Agilent Technologies, 2850 Centerville Road, Wilmington, DE, 19808, USA

Development of GC/MS/MS multiple-reaction monitoring (MRM) transitions is a challenging and time-consuming multi-step process, which may be further complicated by analyte coelution and matrix interferences. A highly automated optimization tool for MRM acquisition, MassHunter Optimizer for GC/TQ, enables optimization of the data acquisition parameters for MRM mode.

End-to-end MRM method development can be highly automated, with no user interaction. Alternatively, each of the optimization steps can be performed individually. These steps include:

- Identification of the analytes using library search of deconvoluted spectra
- Precursor ion identification
- Product ion identification at various collision energies
- Selection of product ions
- Optimization of collision energy

Several workflows available with the Optimizer for GC/TQ, such as start from scan data and start from SIM ions, allow new TQ users to convert existing single quadrupole scan or SIM methods to triple quadrupole MRM methods. Existing TQ users can re-optimize collision energies for current MRMs and update their retention times under new chromatographic conditions with the start with MRMs workflow.

In this work, the Optimizer for GC/TQ was used to efficiently develop up to eighteen transitions for each of the twenty-five pesticides of interest using a pesticide standard mixture in solvent following the start from scan data workflow. Then, the collision energies for the developed MRM transitions were re-optimized in cannabis matrix. The final, matrix-optimized MRM acquisition method included up to five transitions per compound and enabled the highest selectivity for target pesticides in a challenging cannabis matrix. The optimized results were saved as a time segment or a dMRM method.

Thursday, July 29, 2021
11:45 am-12:15 pm
Waters Corporation

Approaches to PFAS Analysis in Food Products

Kari Organtini, Waters Corporation, Milford, MA, USA

Our food supply has many routes of exposure for contamination with per and polyfluoroalkyl substances (PFAS) due to their extensive use and subsequent contamination in the environment. Therefore, it is important to have methods for analyzing both agricultural commodities and food of animal original to better understand and monitor contamination levels. This seminar will discuss different approaches to the extraction of PFAS from various commodities, using QuEChERS, Solid Phase Extraction (SPE), and an automated pressurized fluid extraction (PFE) device, the CEM EDGE, followed by analysis using high sensitivity LC-MS/MS.

Friday, July 29, 2021
12:30-1:00 pm
Bruker Scientific

Higher Sensitivity, Higher Specificity, and Higher Throughput – New Mass Spectrometric Solutions for Chemical Residue Analysis

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

With ever increasing demands for lower detection limits and growing variety of chemical residues that need to be tested for developing a fast, sensitive and accurate screening method is a complex and critical analytical task. To meet these challenging demands, Bruker Scientific has developed several new MS technologies and analytical workflows that will be discussed at this seminar. In particular, a new VIP-HESI ionization source brings the sensitivity of Bruker high-resolution accurate mass QTOF instruments to the levels previously achievable only with nominal mass triple quadrupole systems. An improved Trapped Ion Mobility Spectrometry separation stage in the new timsTOF Pro 2 instrument provides an additional boost to sensitivity in complex matrixes by utilizing collisional cross section filtering to increase the signal to noise ratio. The tandem of high-resolution ion mobility and high-resolution mass spectrometry implemented in the timsTOF platform makes it possible to separate the most challenging coeluting isomers, while speeding up the analysis and increasing the throughput by shortening the chromatographic gradient. These and other new developments will be presented at the seminar and supported by examples from real-life applications.

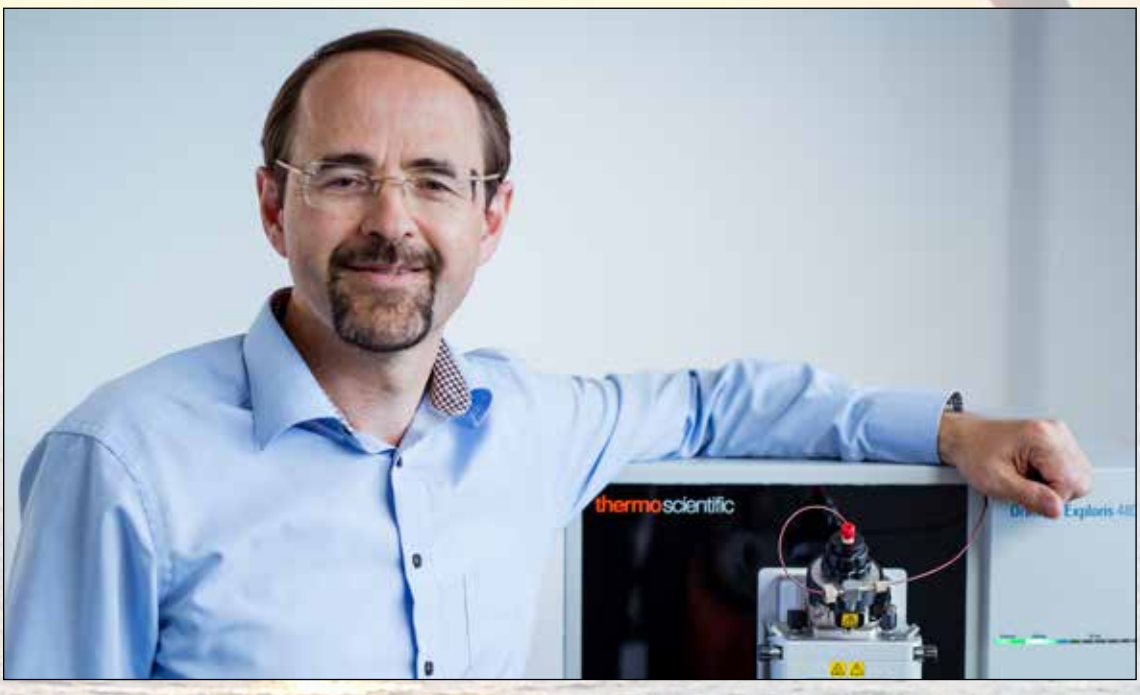
2021 NACRW EXCELLENCE AWARD

PRESENTED TO

Alexander Makarov,
Thermo Fisher Scientific, Bremen, Germany

Alexander Makarov was born in the Siberian town of Irkutsk in 1966 and went to study in Moscow Engineering Physics institute where he also obtained his PhD. After 2 post-doc years at Warwick Univ., he joined a small high-tech company HD Technologies in Manchester (UK).

There he started his work on the Orbitrap mass analyzer. Following the acquisition of the firm by Thermo Electron Corp. in 2000, Alexander provided scientific leadership of the Orbitrap R&D which led to the commercial launch of LTQ Orbitrap mass spectrometer in 2005 and subsequent extensions and new generations of this technology. He has received multiple awards, including Award for Distinguished Contribution in Mass Spectrometry of ASMS and Thomson medal of IMSF. He holds the position of Director of Research, Life Science Mass Spectrometry in Bremen, Germany and Chair in High Resolution Mass Spectrometry at Utrecht University in Netherlands. He was elected a Fellow of the Royal Society (UK) in 2020.



The title of Dr. Makarov's presentation is:

"Exploring New Heights on the Orbitrap Journey"

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MEETING PROGRAM

Monday, July 26, 2021

9:00-10:10 am **Exhibition hour**

10:15-10:45 am **Restek Technology Presentation**
New Technology Solutions for Pesticides Analysis
Joe Konschnik, RESTEK Corporation, Bellefonte, PA, USA

11:00-12:30 pm **NACRW Reference Materials Working Group - RefMaTrA and Beyond!**

The Working Group will present the 1st Edition of "Reference Material Use in Trace Analysis (RefMaTrA)" followed by an Open Forum discussion of a 2nd Edition, including chapters on inorganics and authenticity.

Reference materials and chemical standards are an integral part of the analytical laboratory. NACRW participants and sponsors recognized there was a gap in the availability of current guidance on the use and handling of reference materials and such information was often minimal or provider-focused. The Reference Materials Working Group formed to better gather, sort and characterize information related to calibration standards and other reference materials for both the NACRW and the wider analytical communities. The first edition was created through a collaborative effort by the working group to compile, organize and present best practices focused on organic trace contaminants. In order to further address the needs of the community for contaminants not addressed in the current edition a subsequent edition is now being developed. The current document is being expanded to include topics such as trace metals, inorganics, botanicals, food authenticity, and cannabis reference materials.

11:00-11:20 am **Introduction to the Reference Material Use in Trace Analysis (RefMaTrA) manual**
Joe Konschnik, Restek

11:25- 11:55 am **Suggestions for additional chapters to be added to the 2nd Edition of the RefMaTrA**
Patricia Atkins, SPEX Scientific

12:00-12:30 pm **Discussion (ZOOM Open Forum format)**
Jo Marie Cook, formerly Florida Department of Agriculture

12:40-1:10 pm **SCIEX Technology Presentation**
Highly sensitive quantification and selective identification of pesticides in food with Zeno MRM^{HR}
Robert A. Di Lorenzo, SCIEX, Canada

1:15-3:15 pm **NACRW Veterinary Drugs Working Group proposes collaborative study for regulatory laboratories to determine optimal MS-based analyte identification criteria**

The NACRW Veterinary Drugs Working Group (VDWG) proposes to investigate veterinary drug screening methods which utilize mass spectrometry instrumentation to detect the presence of one or more regulated compounds at levels below the food safety relevant defined maximum levels. At this meeting, the VDWG will describe the details of a proposed regulatory laboratories collaborative study to evaluate currently developed mass spectral methods for the identification of multiple classes of veterinary drugs in several different matrices. Presentations and an Open Forum will discuss optimal-based analyte identification criteria. A collaborative study of multiple matrices and analytes is being planned to evaluate analyte identification criteria to minimize the rate of false positives and false negatives. The Working Group invites those interested in methods meeting the needs of government regulators to share their expertise and suggestions.

1:15-1:45 pm **Optimal MS-based analyte identification techniques**
Steven Lehotay, USDA and **Anton Kaufmann**, Official Control Authority, Switzerland

1:50-2:20 pm **Parameters of proposed collaborative study**
Sherri Turnipseed, FDA and **Eric Verdon**, ANSES, France

2:25-3:15 pm **Questions and Answers (ZOOM Open Forum format)**
Jian Wang, CFIA, and **Jo Marie Cook**, formerly Florida Department of Agriculture

Tuesday, July 27, 2021

9:00-10:00 am **Poster Session (odd number posters only) and Exhibition Hour**

10:00-10:10 am **Opening Remarks**
Sherry Garris, Chair, FLAG Works, Inc.

Sponsored by ThermoFisher Scientific

10:10 am-12 noon **SESSION 1: Award Presentations Session**
Co-Chairs: Mike Filigenzi and Katie Carlos

10:10-10:50 am **Student Poster and Scholarship Awards Presentations**

10:10-10:15 am **Introduction of Scholarship Awards and Student Poster Preview Videos**

10:15-10:22 am **Xinwen Zhang**, University of Delaware, Newark, DE, USA
SP-1 **Developmental toxicity and genotoxicity evaluation of lignin-derivable of six bisguaiacols using in silico, in vitro, and in vivo methods**

10:23-10:30 am **Norma Villagómez-Márquez**, University Of Arizona, Tucson, AZ, USA
SP-2 **Chemicals of emerging concern measured in roof-harvested rainwater to inform environmental justice communities**

10:30-10:37 am **Xin Guo**, University of Massachusetts, Amherst, MA, USA
SP-3 **Mapping microplastics on filter membranes using Raman microscopy: capabilities and limitations in selected food matrices**

10:38- 10:45 am **Jade Noguera**, University of California-Davis, Davis CA, USA
SP-4 **Taking the fat out: Improved sample preparation techniques for toxicologic testing of postmortem liver tissue by GC/MS in veterinary diagnostics**

10:50-10:55 am **Session Sponsor Recognition – ThermoFisher Scientific**

10:55-11:00 am **NACRW Excellence Award Presentation and Keynote Address**
Alexandria Bush, 2021 NACRW Organizing Committee, President

11:00-11:45 am **Presentation by Excellence Award Winner**
Alexander Makarov, Thermo Fisher Scientific, Bremen, Germany
A-1 **Exploring New Heights on the Orbitrap Journey**

11:45-12:00 pm Live Q&A for A-1

12:05- 12:35 pm Thermo Fisher Scientific Technology Presentation
PFAS in NC: Analysis by Isotope Dilution Solid Phase Extraction and LC-MS/MS
Allen Martin, North Carolina Department of Environmental Quality, Raleigh, NC, USA

12:45-12:55 pm Speed Networking with Attendees (groups of 4 for 5 minutes; done twice)

1:00-2:30 pm **SESSION 2: Sample Preparation**
Co-Chairs: Julie Kowalski and Kelly Dorweiler

1:05-1:25 pm **Steven Lehotay**, USDA ARS, Wyndmoor, PA, USA
O-1 Update about the QuEChERSER mega-method

1:25-1:45 pm **Abir Khaled**, University of Waterloo, Waterloo, Ontario, Canada
O-2 Overcoming matrix effects in multi-class multi-residue analysis of veterinary drugs in animal tissue using solid phase microextraction

1:45-2:05 pm **Blair Berger**, University Of Texas at Arlington, Arlington, TX, USA
O-3 Online extraction of microplastics exposed to Polycyclic Aromatic Hydrocarbons (PAHs) using SFE-SFC-MS

2:05-2:30 pm Live Q&A for Session 2

2:30-3:30 pm Poster Session (even number posters only) and Exhibition hour

Wednesday, July 28, 2021

9:00-10:00 am Poster Session (All Posters) and Exhibition Hour

10:00 am-12 noon **SESSION 3: Advancements in QA/QC**
Co-Chairs: Steve Lehotay and Jens Andersen

10:05-10:25 am **Jens Enevold Thaulov Andersen**, Botswana International University of Science and Technology, Palapye, Botswana
O-4 Pooled calibrations with its limitations as an alternative to contemporary methods of QA/QC

10:25-10:45 am **Susan Genualdi**, FDA, College Park, MD, USA
O-5 Challenges in method development and implementation for the analysis of per- and polyfluoroalkyl substances in foods from a QA/QC perspective

10:45-11:05 am **Ivo Leito**, University Of Tartu, Institute Of Chemistry, Tartu, Estonia
O-6 Redefining method validation – ValChrom online tool

11:05-11:25 am **Jacolin Murray**, NIST, Gaithersburg, MD, USA
O-7 Development of a Glyphosate in Oats Reference Material

11:25-12:00 pm Panel discussion of presenters/Live Q&A for Session 3

12:15-12:45 pm **Agilent Technology Presentation**
Automated MRM Method Development for Pesticides Using the Agilent MassHunter Optimizer for GC/TQ

Anastasia A. Andrianova, Agilent Technologies, 2850 Centerville Road, Wilmington, DE, USA

1:00-2:00 pm **SESSION 4: Pesticide Residue Forum**
Moderators: Simon Hird and Brian Eitzer
(Questions can be submitted at registration and through chat)

Session Sponsor Recognition – Restek

Panel of Experts: Steven Lehotay, USDA ARS, Wyndmoor, PA, USA**; André De Kok**, formerly at Wageningen Food Safety Research, Netherlands**; Katerina Mastovska**, Eurofins, Madison, WI, USA**;** and **Jian Wang**, Canadian Food Inspection Agency, Calgary, Alberta, Canada

2:15-4:15 pm **SESSION 5: Cannabis** **Sponsored by Agilent**
Co-Chairs: Yoko Johnson and Kevin Armbrust

2:15-2:20 pm **Session Sponsor Recognition - Agilent**

2:20-2:40 pm **Thuy Vu**, Thuy Vu Consulting LLC, Denver, CO, USA
O-8 **Understanding Food Safety Challenges and Navigating Regulatory Requirements in the Production of Hemp and Cannabis-Derived Compounds**

2:40-3:00 pm **Amy Hernandez**, Louisiana Department Of Agriculture & Forestry, Baton Rouge, LA, USA
O-9 **A Government Lab's Journey into Medical Marijuana and Hemp Analysis**

3:00-3:20 pm **Anthony Macherone**, Agilent & Johns Hopkins SOM, Wilmington, DE, USA
O-10 **Observation of Acid Phytocannabinoid Decarboxylation using Electrospray Ionization in Negative Mode**

3:20-3:40 pm **KC Hyland**, SCIEX, Redwood City, CA, USA
O-11 **LC-MS/MS Sensitivity in the Cannabis Landscape: What Can Be Accomplished with the Latest Generation Triple Quadrupole Platform**

3:40-4:15 pm **Live Q&A for Session 5**

4:15-5:30 pm **Exhibition Hour**

Thursday July 29, 2021

9:00-10:10 am **Exhibition Hour** **Sponsored by SCIEX**

10:15-11:40 am **SESSION 6: Single and Multiresidue Pesticide Methods**
Co-Chairs: Ping Wan and Amadeo Fernández-Alba

10:15-10:20 am **Session Sponsor Recognition - SCIEX**

10:20-10:40 am **Jian Wang**, Canadian Food Inspection Agency, Calgary, Alberta, Canada
O-12 **Applications of nDATA for screening, quantitation and identification of pesticide residues in fruits and vegetables using UHPLC/ESI Q-Orbitrap all ion fragmentation and data independent acquisition**

10:40-11:00 am **Jana Hepner**, Restek, Bellefonte, PA, USA
O-13 **LPGC - The Fast Way to Speed Up Your Multiresidue Pesticide Analysis for Foods!**

11:00-11:20 am **Maria Murcia Morales**, University of Almeria, Almeria, AA, Spain
O-14 **APIStrip, a new tool to assess the environmental pesticide load through honey bee colonies**

11:20-11:40 am **Live Q&A with Session 6 Speakers**

11:45-12:15 pm **Waters Corporation Technology Presentation**
Approaches to PFAS Analysis in Food Products
Kari Organtini, Waters Corporation, Milford, MA, USA

12:20-1:00 pm **Poster Session (All Posters)**

Sponsored by Phenomenex

1:00-2:50 pm **SESSION 7: Perfluorinated Compounds/Environmental Contaminants**
Co-Chairs: Martha Maier and Susie Genualdi

1:00-1:05 pm **Session Sponsor Recognition - Phenomenex**

1:05-1:25 pm **Oliver Cawdell**, Vista Analytical Laboratory, El Dorado Hills, CA, USA
O-15 **PFAS in food matrices: maximizing analytical recovery from important, yet problematic matrices**

1:25-1:45 pm **Lukas Vaclavik**, Eurofins Food Integrity & Innovation, Madison, WI, USA
O-16 **Optimization and validation of an LC-MS/MS method for the determination of PFAS in whole milk, infant formula and related ingredients**

1:45-2:05 pm **Pingping Meng**, North Carolina State University, Raleigh, NC, USA
O-17 **Measurements of novel perfluoroalkyl ether acids (PFEAs) in homegrown blueberries from a PFAS-impacted community in NC**

2:05-2:25 pm **Sara Lupton**, USDA-ARS Edward T. Schafer Agricultural Research Center, Fargo, ND, USA
O-18 **Perfluorooctanoic acid uptake by alfalfa (medicago sativa)**

2:25-2:50 pm **Live Q&A with Session 7 Speakers**

3:00-4:00 pm **Exhibition Hour**

Friday July 30, 2021

Sponsored by Waters Corp.

9:00-10:00 am **Exhibition hour**

10:00-12:20 pm **SESSION 8: New and Advanced Technology in Residue Analysis**
Co-Chairs: Eric Verdon and Sherri Turnipseed

10:00-10:05 am **Session Sponsor Recognition – Waters Corp.**

10:05-10:25 am **Anton Kaufmann**, Official Food Control Authority Of The Canton Of Zurich, Zürich, Switzerland
O-19 **Participating in residue analysis proficiency tests with HRMS; a bold decision?**

10:25-10:45 am **Jeff Archer**, U.S. Food & Drug Administration, Jefferson, AR, USA
O-20 **APGC-QqQ MS, Is it the new gold standard for dioxin and furan determinations?**

10:45-11:05 am **Randall Purves**, Canadian Food Inspection Agency, Saskatoon, Saskatchewan, Canada
O-21 **Using high-field asymmetric waveform ion mobility spectrometry (FAIMS) to improve thyreostatic drug detection in animal tissues using liquid chromatography - selective reaction monitoring (LC-SRM)**

11:05-11:25 am **Michelangelo Anastassiades**, CVUA Stuttgart - EURL-SRM, Fellbach, Bw, Germany
O-22 **Fumigation of dry foodstuff with ethylene oxide – residue problems and measures taken by the EU to tackle the crisis**

11:25-11:45 am **Yelena Sapozhnikova**, Usda, Wyndmoor, PA, USA
O-23 **Screening of chemicals in plastic food contact materials by GC- and LC-Orbitrap mass spectrometry**

11:45 –12:20 pm **Live Q&A for Session 8**

12:30-1:00 pm **Bruker Technology Presentation**

Higher Sensitivity, Higher Specificity, and Higher Throughput – New Mass Spectrometric Solutions for Chemical Residue Analysis

Artem Filipenko, Ph.D., Bruker Scientific, Billerica, MA, US

1:20-3:30 pm **SESSION 9: General Topics**
Co-Chairs: Brittany Holmes and Jessica Krank

1:20-1:40 pm **Yudi Wu**, Florida A&M University, Tallahassee, FL, USA
O-24 **Asses the bioavailability of heavy metal As, Pb and Cr and the phytoremediation potential of Eriocaulon decangulare within the Apalachicola National Forest Basin**

1:45-2:05 pm **Todd Richards**, LECO Corp., Saint Joseph, MI, USA
O-25 **Pesticide detection and quantification in tomato matrix with GCxGC time of flight mass spectrometry**

2:05-2:25 pm **Francisco José Díaz-Galiano**, European Union Reference Laboratory for Pesticide Residues in Fruits & Vegetables and the University Of Almería, La Cañada de San Urbano, Almería, Spain
O-26 **Cutting-edge approach to overcome sensitivity issues associated with polarity switching employing dual-channel chromatography**

2:25-2:45 pm **Ruth Marfil-Vega**, Shimadzu Scientific Instruments, Columbia, MD, USA
O-27 **Essential or precaution? Systematic evaluation of measurements to minimize PFAS background in the laboratory**

2:45-3:15 pm **Live Q&A for Session 9**

3:15-3:30 pm **Poster Awards and Closing**

3:30-4:30 pm **Exhibition Hour**

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ORAL PRESENTATION ABSTRACTS

O-01

Update about the QuEChERSER mega-method

Steven J. Lehotay, Nicolás Michlig, Yelena Sapozhnikova, and Alan Lightfield

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

QuEChERSER is more than QuEChERS in terms of analytical scope of residues and matrices alike (ruggedness), sample throughput, ease of use, automation, efficiency, robustness, and effectiveness (reproducibility). The cost and safety of the two methods are similar, and both are elegant, too. The greatest gains come from combining multiple methods for different applications (*e.g.* veterinary drugs and mycotoxins) into the same method, and from improved sample comminution to reduce the test portion size, which leads to much higher sample throughput by analyzing larger batches. In QuEChERSER, 2 g of thoroughly comminuted sample, ideally using liquid nitrogen, is extracted with 10 mL of 4/1 (v/v) acetonitrile/water for 10 min by shaking, followed by centrifugation for 3 min. A 200 µL portion is transferred to a mini-centrifuge tube and quickly evaporated under nitrogen flow, followed by addition of initial LC mobile phase solvent and ultracentrifuged for 5 min before analysis by UHPLC-(HR)MS(/MS) using alternating dual-columns with backflushing. For GC-amenable analytes, the remaining initial extract is decanted into a 15 mL tube containing 2 g of 4/1 (w/w) MgSO4/NaCl, which is shaken 1 min and centrifuged again for 3 min. Lastly, 1.5 mL of the upper layer is transferred into an autosampler vial, 300 uL of which undergoes cleanup by micro-solid-phase extraction (µSPE) using automated Instrument Top Sample Preparation (ITSP), immediately followed by low pressure (LP) GC-(HR)MS(/MS) analysis. ITSP is conducted in parallel with LPGC-(HR)MS(/MS), which is also conducted in parallel with UHPLC-(HR)MS(/MS), with all methods taking <13 min cycle times per sample. We have begun to evaluate the QuEChERSER mega-method for >500 pesticides, environmental contaminants, veterinary drugs, and mycotoxins in eggs, hemp pellets, and barley. Preliminary findings of the extensive validation study, including a comparison of µSPE+LPGC-HRMS (Orbitrap) with MS/MS results, will be presented.

O-02

Overcoming matrix effects in multi-class multi-residue analysis of veterinary drugs in animal tissue using solid phase microextraction

Abir Khaled[1] and Janusz Pawliszyn[1]

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The current sample preparation techniques used in multiclass multiresidue analysis of veterinary drugs in animal tissue involve time-consuming procedures that are not always effective at minimizing matrix interferences. These methods often involve the use of large amounts of organic solvents which lead to hazardous waste. Moreover, they lack automation and high-throughput capabilities.
This work describes two alternative approaches for multiclass multiresidue analysis of veterinary drugs in animal tissue based on solid phase microextraction. The first approach is based on conventional liquid chromatography- tandem mass spectrometry methods (LC–MS/MS). The second approach is based on the emerging direct analysis MS techniques. In both approaches, the main goal is aimed at minimizing matrix effects and organic solvent use, while maximizing sample throughput.
In the LC-MS/MS based approach, we present a comparison of the developed SPME method to two well-documented sample preparation procedures, namely solvent extraction (SE) and quick, easy, cheap, effective, rugged, and safe (QuEChERS). In the direct to MS approach, we present coupling SPME in different geometries to two different ambient ionization techniques, namely coated blade spray (CBS) and direct analysis in real time (DART). The fully automated sample preparation workflow allows for total extraction time of less than 1 min per sample when 96 extractions are simultaneously conducted, while the direct to MS workflow allows for total analysis time of less than 1 min per sample with screening in both negative and positive ionization modes in the CBS method. All methods were able to achieve excellent accuracy and precision results.

O-03

Online Extraction of Microplastics Exposed to Polycyclic Aromatic Hydrocarbons (PAHs) using SFE-SFC-MS

Blair K. Berger[1], A. Paige Wicker[1], Emily K. Preuss[1], Ayat Omar[1], Alexander S. Kaplitz[1], Brady Drennan[1], Yuka Fujito[2], William Hedgepeth[2], Masayuki Nishimura[2], and Kevin A. Schug;[1]

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Microplastics (µPs) are an emerging global concern, as recycling rates lag behind worldwide plastic production. Plastics enter our environment from a multitude of sources. A variety of physical and environmental factors drive the degradation of larger plastics to smaller particles. Between 5 mm and 1.0 µm in size, µPs have increased surface area; promoting the adsorption, concentration and ultimately the bio-magnification of environmental contaminants. Microplastics research is ample with small sample size challenges and complications due to the variety of environmental pollutants and special sample prep considerations. Standard extraction methods are excessively time consuming and/or require large sample volumes. In this work, online extractions were performed using supercritical fluid extraction – supercritical fluid chromatography – mass spectrometry (SFE-SFC-MS), significantly reducing sample volume, minimizing prep, and increasing throughput. Reference materials (µP-RMs) were cryomilled from three types of common plastics: (PETE, LDPE and HDPE), exposed to marine inter-coastal waters off the U.S. Gulf Coast of Florida and collected after 1, 7 & 14 days for online extraction. Laboratory controls were created for comparison by exposing the same µP-RMs to 50 mL exposure solutions of fresh- or salt-water spiked with polycyclic aromatic hydrocarbons (PAHs) at low (500 ng/g) and high (2,500 ng/g) concentrations, and shaken under conditions mimicking that of the marine exposure (50 rpm; 32 ºC; 1, 7 & 14 days). A pre-developed method for the online extraction of PAHs from soil was used, with minimal modification, allowing rapid online extraction, separation and detection of eleven PAHs directly from pollutant exposed-microplastics.

O-04

Pooled Calibrations with Its Limitations as an Alternative to Contemporary Methods of QA/QC

Jens E.T. Andersen, Keaboletse Moemedi, Kebabonye Katse

Botswana International University of Science and Technology (BIUST), Faculty of Sciences, Department of Chemical and Forensic Sciences, Plot 10071, Boseja Ward, Private Bag 016, Palapye, Botswana; andersenj@biust.ac.bw

Only a few useful recommendations are available, as to the determination of the linear range of calibration. The unfeasible determination of the linear range is evaluated by the corresponding coefficient of regression that depends strongly on the number of data points to the calibration line. The limit of quantitation (LOQ) depends on the standard deviation of blanks that remains highly unreliable, owing to the only ten repetitions that are recommended for its determination. With the new system of QA/QC, that was developed and adopted by numerous national and international organizations, there is more focus on uncertainty of measurement, traceability and uncertainty budgets that allows for production of reliable predictions and delivery of consensus values. Despite good intentions and much effort, the new system of QA/QC is overly complicated and there are some serious shortcomings that need to be addressed. For a method validation with results of Flame-atomic absorption spectrometry equipped with a continuous source lamp, as an example, it is demonstrated that surprisingly large uncertainties were obtained, which contradicted the information that was provided by the manufacturer. By introducing the principle of pooled calibrations (PoPC) the elements Na(588 nm), K(766 nm), Mg(285 nm), Fe(248 nm), Ni(232 nm) and Cu(324 nm) were subjected to method validations. Generally, the lower limit of analysis (LLA) was found to be larger by more than an order of magnitude in comparison with the corresponding LOQ. The uncertainty of measurement as a function of concentration reached high levels thus effectively narrowing the analytical range of concentrations.

O-05

Challenges in Method Development and Implementation for the Analysis of Per- and Polyfluoroalkyl Substances in Foods from a QA/QC perspective

Susan Genualdi, Wendy Young, Lowri deJager, Timothy Begley

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The development of analytical methods for per- and polyfluoroalkyl substances (PFAS) in a wide variety of foods and agricultural products is necessary for understanding their contribution to the human diet. During the method development of composite table-ready food samples for 16 different PFAS chemicals, challenges arose with matrix suppression, interferences, false positives, and method blanks. Secondary clean-up steps were useful in the removal of some matrix interferences, but some PFAS have only one MRM transition, which makes the positive identification of these analytes challenging. Further investigations were made into differences in relative retention times, column length, and confirmation using exact mass instruments. Blanks are a known challenge with PFAS analysis due the use of fluorinated polymers in the lab environment. Concentrations of lab blanks are presented and how samples are corrected is described. This method was further applied to various agricultural samples and challenges with these matrices are presented and discussed.

O-06

Redefining method validation – ValChrom online tool

Asko Laaniste,[1] Koit Herodes,[1] and Ivo Leito[1]

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This presentation is about an education-related activity in process here at UT: a web-based validation tool – ValChrom (https://sisu.ut.ee/lcms_method_validation/14-valchrom) – which on one hand teaches how validation (first of all for chromatographic methods) should be done and on the other hand helps professionals with experiment planning and calculations of performance parameters in order to reduce the time and effort involved.

Important abilities and features of ValChrom are:
(1) ValChrom enables validation according to specific Guideline documents, e.g., Eurachem, EMA, ICH, … and combination of requirements from different guidelines.
(2) ValChrom „walks the user through validation“ of his/her specific method and automatically creates the experiment plan on the basis of information about method parameters and requirements entered by the user. The user will have a good overview of all the performance parameters and possibility to generate detailed reports.
(3) ValChrom is web-based, centrally managed and aimed to be “community checked” for quality. It is accessible from anywhere and is free of charge.

At this stage, ValChrom is still a work in progress, but sufficient amount of functionality is already available, so that it can already be used as a tool for teaching and also offers great help for actual validation experiments. Using ValChrom is free of charge and user account can be created at the above address. We expect that ValChrom could be interesting for a widest range of labs doing LC or LC-MS analysis.

O-07

Development of a Glyphosate in Oats Reference Material

Jacolin A. Murray[1], Justine M. Cruz[1], Andrea J. Yarberry[1], Graham Yearwood[2], Katrice A. Lippa[1]

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Proper quality assurance/quality control (QA/QC) practices are important to ensure reliability of results in the analysis of pesticide residues in food. Reference materials (RMs) that are similar to the matrix and residue levels to the samples being analyzed can be useful as QC materials in the evaluation of potential method biases. In addition, RMs containing incurred analytes are important in evaluating extraction efficiency, however, there are a limited number of available RMs for such applications. Due to the high occurrence of glyphosate residues in food, many stakeholders have expressed an interest for a glyphosate in cereal grain-based RM. The National Institute of Standards and Technology (NIST) is currently developing a two-level glyphosate in oat material suite. In order to identify potential RM candidates, several types of oat-based cereals and oat flours were screened for glyphosate and its major metabolite aminomethylphosphonic acid (AMPA) by LC-MS/MS after derivatizing with 9-fluroenylmethyl-chloroformate (FMOC-Cl). After initial screening, several oat flours were selected and blended to produce both a “high” and a “low” level glyphosate candidate material. A homogeneity study was performed after the materials were packaged. An extensive stability study was performed at different storage conditions to evaluate long term stability of glyphosate in an oat matrix. The candidate RMs were also screened in an interlaboratory study to determine if the materials are fit-for-purpose for routine QC applications in food testing laboratories.

O-08

Understanding Food Safety Challenges and Navigating Regulatory Requirements in the Production of Hemp and Cannabis-Derived Compounds

Thuy Vu

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The passing of the 2018 Farm Bill has fueled competitive innovation of unique cannabidiol-infused products amongst manufacturers from introducing hemp-derived cannabinoids into products for oral ingestion, inhalation, and topical application as well as pet products. The creation of novel equipment and techniques as well as the adoption of technological applications from other industries out of necessity has given rise to a myriad of hemp-derived compounds, from full-spectrum, broad-spectrum, distillates, isolated cannabinoids, and terpenes. THC-remediation and cannabinoid conversions have afforded the industry pathways to meet regulatory requirements, utilize all components of the hemp plant, and minimize waste streams. How do regulators keep up with addressing food safety challenges? How does the industry navigate regulatory requirements that may vary from state to state without clear direction from the federal government? We will briefly explore food safety considerations and how to proactively address regulatory requirements during the manufacturing of hemp-derived compounds, intermediate hemp products, and finished hemp products.

O-09

A Government Lab’s Journey into Medical Marijuana and Hemp Analysis

Amy B. Hernandez
(Marijuana Team Members: Emily Harrelson, Jessica Landry, Buffy Meyer, Chantilly Reddmann, Andrea Warren)

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Follow the Louisiana Department of Agriculture & Forestry briefly through the process of learning about medical marijuana regulations, establishing state regulations and then developing methods to analyze samples against their regulations. Louisiana regulates medical marijuana for potency and homogeneity, microbiological contaminants, heavy metals, residual solvents, mycotoxins and pesticides. The main focus of the presentation will be the analytical methods currently in use by the laboratory. Difficulties encountered especially in pesticide analysis will be shared. Currently, the lab is building on these experiences and the experiences of the great network of agricultural labs in the United States to develop a hemp analysis program. The lab's current plans will be shared along with outstanding issues and anticipated problems.
Hind sight is 20/20 and all that jazz!

O-10

Observation of Acid Phytocannabinoid Decarboxylation using Electrospray Ionization in Negative Mode

Anthony Macherone[1,2], Peter J.W. Stone[1], Sue D'Antonio[1], Nikolas C. Lau[1], Wendi A. Hale[1].

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Acid phytocannabinoids are present in the living *Cannabis spp*. plant at much higher concentrations than their decarboxylated analogues. Δ^9-tetrahydrocannabinolic acid (THCA) and cannabidiolic acid (CBDA) are enzymatically biosynthesized from cannabigerolic acid (CBGA) through the action of THCA-synthase and CBDA-synthase, respectively. Decarboxylation of THCA to psychoactive Δ^9-tetrahydrocannabinol (THC), and CBDA to non-psychoactive cannabidiol (CBD) occurs primarily post-harvest through exposure to light and heat. Decarboxylation also occurs when chemical analysis is performed with GC or GC/MS systems. In this study, we evaluated the stability of acid phytocannabinoids analyzed with positive and negative electrospray (ESI+ and ESI-, respectively) LC/MS as a function of drying gas temperature. In ESI+ mode, post-chromatographic (in-source) acid decarboxylation was not observed over a drying gas temperature range of 75°C through 300°C. However, ESI- mode experiments did reveal decarboxylation of THCA and CBDA and increasing relative abundances of THC and CBD with increasing drying gas temperatures. These data highlight a potential concern for the analyses of acid phytocannabinoids using ESI- mode LC/MS.

O-11

LC-MS/MS Sensitivity in the Cannabis Landscape: What Can Be Accomplished with the Newest Triple Quad

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"Cannabis testing" is a commonplace phrase which in fact encompasses multiple and diverse assays, regulations, compliance requirements, method challenges and data quality needs.

Compliance requirements around pesticide residues are varied among regions for which regulations exist. The trend with time and legalizing legislation seems to be towards increasingly rigid requirements (i.e., more pesticides and lower detection limits). Anticipation of increasingly aggressive analytical requirements necessitates development of pesticide quantification methods which are as sensitive and robust as possible. Simultaneously, forensic and toxicology labs are faced with increased need for rapid and sensitive determination of cannabis consumption in monitoring patient samples.

The SCIEX 7500 LC-MS system provides gains in sensitivity over previous triple quadrupole platforms by improving ion generation and sampling more ions; improvements in detection limits were observed for the Canadian panel of pesticides in Cannabis products. Additional sensitivity allows use of larger sample dilutions, protecting the front end from contamination between routine cleanings (robustness), and provides flexibility to adapt to tightening regulations.
In addition to advancing pesticide quantification, an MS3 workflow leveraged the instrument's ion trap functionality to successfully eliminate background and matrix interferences at very low levels, improving the selectivity (and subsequently, the sensitivity) of the cannabinoid THC-COOH in oral fluid.

O-12

Applications of nDATA for screening, quantitation and identification of pesticide residues in fruits and vegetables using UHPLC/ESI Q-Orbitrap all ion fragmentation and data independent acquisition

Jian Wang[1], Willis Chow[1], Jon W. Wong[2], James Chang[3]

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High sample throughput and effective multi-residue methods for screening, quantitation and identification are desired for the analysis of a large number of pesticides in routine monitoring programs for food safety. This study was to explore the use of UHPLC/ESI Q-Orbitrap (or UHPLC/ESI, thereafter, if not specifically denoted) non-target data acquisition for target analysis (nDATA) for the screening a large group of less frequently found pesticides while quantitating and identifying a group of fifty frequently incurred residues in fruits and vegetables in a single analysis. High-resolution mass spectrometry such as Q-Orbitrap has unique applications for pesticide analysis because of its full scan capability along with data independent acquisition (DIA) or all ion fragmentation (AIF). The experiments were designed to achieve a balance between selectivity and cycle time for screening of 655 pesticides while quantifying and identifying 50 pesticides in fruits and vegetables by considering the parameter settings such as mass resolution and the number of mass isolation windows or isolation window widths. Coupled with ultra-high performance liquid chromatography, both Full MS/DIA and Full MS/AIF allowed this nDATA workflow to perform screening, quantification and identification in a single analysis. In general, UHPLC/ESI Full MS/vDIA detected more fragment ions of the pesticide than AIF in all scenarios when one to four fragments were compared. UHPLC/ESI Full MS/vDIA and AIF generated comparable quantitative results but the latter provided better precision due to it shorter cycle time or more scans across a chromatographic peak. UHPLC/ESI Full MS/vDIA may be preferable to be used for screening, quantitation and identification of pesticides, especially when the testing scope covers a large group of compounds, for example over hundreds of pesticides.

O-13

LPGC - The Fast Way to Speed Up Your Multiresidue Pesticide Analysis for Foods!

Jana R Hepner,[1] Jaap deZeeuw,[1] Kristi Sellers,[1] and Joseph Konschnik[1]

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Food testing labs need fast turn-around times to analyze samples submitted for multiresidue pesticide analysis and deliver accurate results. Now, they can take advantage of their mass spectrometer's vacuum to give their gas chromatography workflow a significant speed boost. Installing a new factory-coupled column set allows the vacuum in the MS detector to lower the pressure inside the analytical GC column, which results in run times three, or more times faster than a conventional GC-MS column configuration. The technique is called low-pressure gas chromatography (LPGC). Given the challenges with making difficult column connections, this technique has lacked adoption in the past. The authors present data showing a typical GC-MS/MS multiresidue pesticide workflow for various food commodities using a conventional 30 meter column compared with the faster analysis using this innovative pre-connected column set to demonstrate the ease and effectiveness of implementing this technique. A comparison will be made between the two identifying the benefits and challenges of this and other alternative fast GC approaches.

O-14

APIStrip, a new tool to assess the environmental pesticide load through honey bee colonies

Maria Murcia-Morales[1], Francisco José Díaz-Galiano[1], José Manuel Flores[2], Jozef J. M. Van der Steen[3], Amadeo R. Fernández-Alba[1]

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[2]Department of Zoology, University of Córdoba, Campus of Rabanales, 14071, Córdoba, Spain
[3]Alveus AB Consultancy, Oisterwijk, the Netherlands

Honey bees are exposed to pesticides and other contaminants during their foraging activities, in which they are in close contact with crops in a ratio of several kilometers from their beehives. Hence, the analysis of beehive matrices can be a useful approach for the assessment of environmental contamination. However, the classical active sampling strategies involve an alteration of the colonies due to the need of taking biological matrices (wax, living bees, bee bread or honey, among others), which makes it difficult to perform long monitoring studies. Additionally, an efficient evaluation of the contamination pressure inside the beehive has not been achieved until now, due to the different nature of the multiple matrices where contaminants can accumulate. In this context, the APIStrip-based passive sampling approach minimizes the human interaction with the colonies, allows for multiple subsequent samplings in one single beehive and provides comprehensive information about the contaminants present in the colony environment. APIStrips are based on the sorbent Tenax, one of the most versatile adsorbents used for the passive sampling of environmental contaminants, and these passive sampling devices are currently being employed in a pilot monitoring program in nine European countries (INSIGNIA project). In the present communication, the preparation and optimization procedure, extraction method and analysis for the APIStrips will be described. The obtained results for their application in different field studies (aimed at assessing the dissipation of some common miticides as well as the environmental contamination) will also be discussed.

O-15

PFAS in Food Matrices: Maximizing analytical recovery from important, yet problematic matrices

Oliver Cawdell and Martha Maier

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Per- and polyfluoroalkyl substances (PFAS) are increasingly the subject of environmental research because they are highly mobile in the environment, ubiquitous, bio-accumulative and associated with various adverse long-term health effects. For these reasons, the detection limits for PFAS must be set very low to account for a lifetime exposure. However, it is difficult to achieve low detection limits for complex matrices with variable interferences.

Whilst most research focuses on drinking water as the primary exposure pathway, understanding and quantifying PFAS in food remains an important area for public health. Developing a method which can effectively extract PFAS from food matrices can be challenging. Interference sources such as proteins, lipids and high organic matter content can disrupt PFAS recovery. Understanding the limitations of different method approaches is key for validating food matrix results. This study compares the QuEChERS technique to a standard Solid Phase Extraction (SPE), with cleanup methods for varying types of food samples.

Dried and fresh corn, minced beef, chicken egg, natural yogurt and vegetable oil samples were obtained from a grocery store. One gram of each matrix was extracted using a basic pre-treatment methanol extraction and QuEChERS extraction technique. Then these extracts were processed using a range of cleanup options to assess the combination of approaches that best mitigate food matrix interferences. Column and dispersive-SPE, Envi-Carb and a selective lipid removal SPE were compared. All samples were analyzed via UPLC-MS/MS with an isotope dilution technique.

O-16

Optimization and Validation of an LC-MS/MS Method for the Determination of PFAS in Whole Milk, Infant Formula and Related Ingredients

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Per- and polyfluoroalkyl substances (PFASs) are a wide group of anthropogenic chemicals that have been applied in a wide range of industrial, commercial and domestic products since the 1950s. Due to their toxicity, persistence and bioaccumulation potential, PFAS 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 respect, whole milk, infant formula and ingredients used in infant formula production represent important matrices that require sensitive methods with reporting limits at low parts per billion levels or lower. In this study, an LC-MS/MS method was optimized and thoroughly validated for the low-level determination of sixteen priority PFAS analytes listed by the US Food and Drug Administration (U.S. FDA). The presented method was based on a QuEChERS procedure developed and recently published by the U.S. FDA and further improved to expand matrix scope and increase sample throughput. Acceptable performance in terms of selectivity, method detection limit, limit of quantification, accuracy and precision was obtained for all analytes with spike recoveries within 70-120 % and intermediate precision (RSD_{INT}) below 20%. The presentation will summarize background, currently available methodologies, optimization experiments, strategies to eliminate & avoid PFAS background contamination, as well as the outcomes of the method validation in whole milk, milk-based powdered infant formula, ready-to-feed infant formula and ingredients, such as milk powder, maltodextrin and vegetable oil.

O-17

Measurements of Novel Perfluoroalkyl Ether Acids (PFEAs) in Homegrown Blueberries from a PFAS-Impacted Community in North Carolina

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In the Cape Fear River basin of North Carolina, novel perfluoroalkyl ether acids (PFEAs) such as hexafluoropropylene oxide-dimer acid (commonly known as GenX) have received public and regulatory attention due to their detection at high levels in private wells and public water systems. Apart from drinking water, diet may be another important route of exposure. Many people in the impacted community grow fruits and vegetables in their gardens, but the uptake of PFEAs and legacy per- and polyfluoroalkyl substances (PFASs) into local produce remains unclear. Extraction methods exist for legacy PFASs, but their suitability for novel PFEAs requires further investigation. In this study, an extraction and clean-up method was developed for the quantification of 44 PFASs, including 14 PFEAs, in blueberry and blackberry samples. The extraction workflow includes homogenization and extraction using 0.01 M ammonium hydroxide in methanol. Mass-labeled internal standards were added prior to extraction, which included vortex, ultra-sonication and centrifugation for a total of three times, followed by a SPE purification using Oasis WAX cartridges. In method validation, satisfactory performance was achieved with recoveries of 30 PFASs (including 9 PFEAs) within 70-130% at 2 spike levels. The recoveries of 12 PFEAs were within 50-200%. Homegrown blueberries from a PFAS-impacted Community were extracted and analyzed using liquid chromatography−tandem mass spectrometry (LC−MS/MS). Novel PFEAs were detected with concentrations ranging from 0.13 to 7.24 ng/g wet weight. Blueberries showed elevated levels of perfluoromethoxyacetic acid (PFMOAA), perfluoro-2-methoxypropanoic acid (PMPA), and perfluorodioxahexanoic acid (PFO2HxA) at 1.81, 3.80 and 7.24 ng/g wet weight respectively.

O-18

Perfluorooctanoic Acid Uptake by Alfalfa (*Medicago sativa*)

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Perfluorooctanoic acid (PFOA) is a perfluorinated alkyl substance (PFAS) used as a surfactant in industrial and consumer products such as coatings for paper and textile fabrics but also is a degradation product from other PFAS formulations. Concern has increased over presence of PFOA in wastewater treatment plants and biosolids from these plants which are used as fertilizer on agricultural lands, additionally PFOA contamination has been found in aquifers due to use of firefighting foams near airports and Air Force bases. Use of biosolids and contaminated water on agricultural land could result in release of PFOA into the environment accompanied by transport through the soil and/or uptake by agricultural crops, such as alfalfa, that are used to feed livestock. A study was undertaken to quantify the uptake by alfalfa and transport through soil of ^{14}C-PFOA topically applied to a typical North Dakota agricultural soil over 10 weeks. By 7 days post application, PFOA was measurable in the roots, stems, and leaves with accumulation being the greatest in the leaves over the 10 weeks. PFOA migration over 10 weeks through unplanted soil reached a depth of 22.8 ± 2.5 cm, whereas in planted soil PFOA only migrated to a depth of 7.5 ± 2.5 cm. PFOA transport through soil may be deterred by the presence of crops, however PFOA accumulation in the edible portions of the crops may increase food animal exposure to PFOA residues and also possibly contaminate further consumer products.

O-19

Participating in residue analysis proficiency tests with HRMS; a bold decision?

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Laboratories working in the field of residue analysis (e.g. pesticides, veterinary drugs and environmental contaminants) in complex matrices are required to regularly participate in proficiency tests (PT) to prove their capability of detecting and accurately quantifying residues of interest. Routine residue analysis is still overwhelmingly dominated by tandem quadrupole instrumentation (QqQ) which has been termed the “gold standard”. Considering the importance of receiving good PT scores, is it wise to analyse an unknown PT sample with high resolution mass spectrometry (HRMS), while dozens of other participating laboratories use their time proven QqQ instrumentation? This question shall be answered by comparing the outcomes of the more than 100 different PT tests in which we have participated during a period of nearly 10 years.

O-20

APGC-QqQ MS, Is It the New Gold Standard for Dioxin and Furan Determinations?

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The use of High-Resolution Gas Chromatography / High Resolution Mass Spectrometry (HRGC/HRMS) has been well established as the “Gold Standard” for the determination Dioxins and Furans (D/Fs) for more than 40 years. Magnetic sector HRMS instruments were used for development of EPA Method 1613, “Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS,” using an isotope dilution technique and collecting data via Selected Ion Monitoring (SIM). The current EPA Method (Revision B, 1994) still requires HRMS instrumentation due to the needed sensitivity and selectivity. Alternative instrumentation has been investigated over the past several years due to manufacturers’ decisions to phase-out magnetic sector instrument production. A European Union directive (Commission

Regulation (EU) No 589/2014 of 2 June 2014) states that a triple quadrupole mass spectrometer (GC-QqQ MS) could be used for D/F and PCB confirmation, provided sensitivity requirements are met (measuring at or below 1/5 of the tolerance level) with acceptable ion ratios. We have performed an instrument platform extension for D/F determinations using an Atmospheric Pressure Chemical Ionization Gas Chromatograph (APGC) QqQ MS by comparing HRMS side-by-side results from 24 extracts and will show precision and accuracy data comparing these platforms. Additional data showing results from international interlaboratory studies will also be discussed.

O-21

Using high-field asymmetric waveform ion mobility spectrometry (FAIMS) to improve thyreostatic drug detection in animal tissues using liquid chromatography – selective reaction monitoring (LC-SRM)

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Thyreostatic drugs (thyreostats) interfere with thyroid function and have been used illegally in animals slaughtered for food. Thyreostat use leads to a poorer quality meat due to increased water retention and the residues of these drugs can also lead to adverse effects in humans. Since these drugs, with the exception of thiouracil (TU), do not occur naturally, sensitive methods are required for their detection in animal tissues. Although LC-SRM is typically used, difficulties arise because thyreostats are low molecular weight (114-204), polar analytes and LC-SRM detection limits suffer from high chemical background and endogenous interferences. Differential ion mobility techniques separate gas-phase ions and we implemented a high-field asymmetric waveform ion mobility spectrometry (FAIMS) interface in combination with LC-SRM to reduce chemical background. Optimum values of transmission through the FAIMS interface were established for six thyreostats and transmission of background ions was observed to occur at different values. Using the same conditions, LC-FAIMS-SRM results were generated and compared with the results of our validated LC-SRM method. For the most problematic thyreostats, such as tapazole (TAP), improvements in S/N of up to 50 times were observed. This resulted in wider linear ranges with the lower limit of quantification being up to 20 times lower (0.05 ppb for TAP and methylthiouracil (MTU) by LC-FAIMS-SRM compared with 1 ppb with LC-SRM). For phenylthiouracil (PhTU) the improvements were more modest (S/N ~2 times better) since chemical background was not as prevalent. Overall, the LC-FAIMS-SRM approach showed significant improvements, particularly for analytes with high chemical backgrounds.

O-22

Fumigation of Dry Foodstuff with Ethylene Oxide – Residue Problems and Measures taken by the EU to Tackle the Crisis

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Ethylene oxide (EO) is used to produce detergents, thickeners and plastics but also for fumigations of medical equipment, tobacco, textiles and dry food. After fumigation, food is aerated but some EO previously reacts with matrix-components giving e.g. glycol, 2-chloroethanol (2CE) and hydroxyethyl-adducts of matrix-components. So, EO is virtually never detected in food. Its reactivity makes EO hazardous (1B-carcinogen), and EO-fumigations of food were banned in EU 30 year ago. EU-MRLs were set at LOQ with the RD including 2CE.

The German official method involves transformation of 2CE to EO, conversion to 2-iodoethanol, partitioning to ethyl-acetate and GC-analysis. The method is complex and time-consuming and recoveries are moderate, so it was only implemented by few labs, but due to rare findings, it was eventually abandoned by most. EO was thus rarely controlled in the EU until September 2020, when a RASFF-warning on EO in Indian sesame at levels >3000-fold the EU-MRL was released. Hundreds RASFF-notifications on EO in sesame and other products followed. But how come

that EO-fumigations suddenly became so massive? This trend is linked to an EU-requirement for sesame-imports to be accompanied by certificates proving Salmonella absence, to prevent salmonellosis-outbreaks.

To facilitate 2CE/EO analysis, EURL-SRM developed a method allowing quick analysis of EO and 2CE involving QuEChERS- or QuOil-extraction, dSPE-cleanup (PSA/C18) and GC-MS/MS. Recoveries typically exceed 85%. The separate analysis of EO and 2CE allows differentiated risk-assessment. The EURL-SRM, furthermore, organized 2 PTs so that labs can demonstrate performance. An overview of residue-findings in various matrices will be shown.

O-23

Screening of chemicals in plastic food contact materials by GC- and LC-Orbitrap mass spectrometry

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It is estimated that more than 10,000 chemicals are intentionally used in the manufacturing of food contact materials (FCMs), a group of chemicals called intentionally added substances (IAS). However, the information on the identity, potential migration into foods and effect on human health of these chemicals lacking. Moreover, many non-intentionally added substances (NIAS) are formed during the manufacturing, storage and use, and even less is known about NIAS. The goal of this study was to apply non-targeted analysis with gas and liquid chromatography high resolution mass spectrometry (HRMS) Orbitrap MS to identify chemicals migrating from plastic FCMs during microwave and conventional oven treatments. Migration experiments utilizing food simulant were conducted for samples of microwave meal trays, microwave bags and oven bags. Identification of migrated compounds was performed with existing HRMS databases for GC- and LC-HRMS with mass error <5 ppm, and 74 migrated compounds were identified. Of these, 65 were IAS or chemicals associated with production of polymers and plastics. Another 9 migrated chemicals were NIAS, including oxidized products and derivatives of commonly used food contact substances. Increased migration of chemicals for microwave compared to conventional oven treatments was observed, where 20 migrated chemicals had significantly higher levels (p<0.05) for microwave vs. conventional oven treatment. For several identified chemicals, no previous reports on their migration from FCMs were found.

O-24

Assess the bioavailability of heavy metal As, Pb and Cr and the phytoremediation potential of *Eriocaulon decangulare* within the Apalachicola National Forest Basin

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Heavy metal contamination in soil is one of the most significant environmental problems. In this study, a field study was carried out to estimate the effect of the plant phytoremediation and rhizosphere microorganism community on heavy metal remediation within the Apalachicola national river basin. Forest soil was found polluted by heavy metals including As, Pb and Cr to different extent. Specifically, up to 660.68 ± 22.66 mg/g As has been retained in the bulk soil of study region. Potential toxicity of three heavy metal has been estimated by the bioavailability to the surrounding microorganism and plants. In normal bulk soil, bioavailability indicators of three heavy metal are relatively high and has a sequence of Cr (38.04) > Pb (24.98) > As (4.90). Bioavailability of three heavy metals has been decreased up to 76. 24% in the rhizosphere soil where plant root located. Plant and rhizosphere microorganism played important roles during this process. *Eriocaulon decangulare* is the native species that ubiquitous in the Apalachicola national forest. It is functional in two major ways, phytoextraction and phytoimmobilization. EDS mapping successfully located accumulated heavy metal on aerial part and roots.
Microorganism community analysis indicates the major functional bacteria species include *Proteobacteria, Acidobacteria* and *Verrucomicrobiota*. In conclusion, results indicated As pollution in the Apalachicola river basin should be taken particular concern. *Eriocaulon decangulare* could be a potential phytoremediator for the future pollution control.

O-25

Pesticide Detection and Quantification in Tomato Matrix with GCxGC Time of Flight Mass Spectrometry

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Pesticide detection and quantitation in food matrices continues to be an ongoing challenge. Typically the primary hurdle to this analysis is the matrix itself. While significant improvements have been made to extraction and cleanup methodologies of the sample matrix, interferences persist which inhibit detection and may skew quantitative accuracy. Leveraging the additional chromatographic resolution of GCxGC we are able to separate target pesticides from matrix and other pesticides that would typically co-elute with standard, single dimension GC.

GC×GC/MS analysis of matrix matched standards was performed using a LECO Pegasus BT 4D high performance time of flight mass spectrometer (TOF-MS) coupled with an Agilent 7890 fitted with a GL Sciences cryotrap inlet. Data were acquired from 40-600m/z at 250 spectra/second. During data review all standards and guidelines for nominal resolution, TOF-MS systems outlined in SANTE 2017 were followed to ensure identification and quantitative accuracy. Quantitation curves for each of the pesticides were created and used to quantify their levels in an unknown, spiked tomato matrix extract. The presentation will focus on how GCxGC mitigated matrix interferences with target compounds leading to improved quantitation accuracy and limits of detection.

O-26

Cutting-edge approach to overcome sensitivity issues associated with polarity switching employing dual-channel chromatography

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Optimal mobile phase modifiers are not equivalent for positive and negative ionisation modes in electrospray. For this reason, it is complicated to achieve high sensitivity in multiresidue methods which combine the analysis of compounds which ionise in the positive and negative polarity modes. This issue can be overcome by independently injecting a sample twice. In the first injection, the sample is analysed employing a mobile phase optimised for positive mode electrospray ionisation, which contains water, methanol, formic acid, and ammonium formate; whereas during the second injection, a mobile phase negative mode optimised for electrospray ionisation, which contains water, acetonitrile, and acetic acid is used. A fast analytical approach has been validated for the analysis of pesticide residues in baby food at low concentrations based on the previously described method. After an acetonitrile-based extraction, samples were analysed with a dual- channel liquid chromatography instrument coupled to a triple quadrupole mass spectrometer. The number of evaluated pesticides was 264, from which 238 were analysed with the methanol-based gradient and 26 with the acetonitrile-based gradient. The dual-channel instrument allowed for sample multiplexing, thus, sample analysis in duplicate (by both gradients) was faster than one injection using a single-channel system, 14.35 min and 18 min, respectively. The limit of quantitation of the proposed method was determined according to the DG SANTE Document, which was set to 0.003 mg/kg for 97 % of the compounds. The validation study was followed by a real 42 baby food sample survey, during which 16 positive results were detected.

O-27

Essential or precaution? Systematic evaluation of measurements to minimize PFAS background in the laboratory

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Interest in the analysis of Per- and Polyfluoroalkyl Substances (PFAS) in environmental, food and consumer products samples has dramatically increased in the last five years. However, scientists from various research fields have been working on studying these chemicals for decades. During this time, numerous technological advancements have been introduced in the analytical instrumentation devoted to the analysis of PFAS and other trace contaminants to improve sensitivity, ease the overall workflows... One aspect that remains practically unchanged since the early years of PFAS research is related modifying the LC-MS/MS configuration. Replumbing the LC-MS/MS and replacing additional components were implemented to minimize PFAS background contamination during the analysis. Some of these modifications are not user-friendly, require the purchase of additional supplies for the laboratory and sometimes delay the generation of results. Though, are these measurements essential or are just precautionary steps implemented years ago without further evaluation? In this presentation we will share the outcomes from the systematic evaluation of several variables affecting PFAS analysis by LC-MS/MS (including hardware configuration, consumables…) and provide an overview of which modifications could be avoided.

water and sediment, conducted from february to June 2018; for which it was using liquid scintillation spectrometry to realize the balance of mass of the applied product. Determined the rate of absorption (k1), the depuration rate (k2) and the half-lives biological (t1/2) that is 0.65 L kg-1 d-1; 0.16 d-1 and 4.33 d respectively. It was found after the phase of exposure in trout, water and sediment 359,4 ng g-1, 3527,32 ng l-1, and 344,79 ng g-1 respectively and after the depuration phase 131,67 ng g-1, 539,43 ng l-1 and 32,24 ng g-1 respectively.

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

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Due to the environmental persistence of PFOA, PFOS, and other similar compounds, industrial and commercial fluorochemical manufacturers have started producing alternatives. These alternatives have been identified as ether forms of PFOA and other similar compounds. One of these new compounds is referred to as GenX, with the chemical name HFPO-DA. This compound has been detected in the environment at multiple contaminated sites and has been added to the EPA 537.1 method. It is important for any PFAS testing lab to include these compounds in their protocols. In this study, different LC/MS/MS workflows to detect and quantitate HFPO-DA, DONA, and ADONA, in water and soil samples were evaluated. The use of a Scheduled MRM algorithm using a SCIEX QTRAP® system that automatically triggers secondary MRM transitions, for increased data quality and confidence in analyte detection at low ng/mL concentration levels is described; as well as the use of a quantitative High Resolution MRM workflow using a SCIEX QTOF system to provide high-resolution, accurate-mass data for full-scan information of both precursor ion and all product ions.
The methodology presented also includes 24 other commonly tested PFAS using two different sample preparation approaches: The first was a dilute-and-shoot method that used an optimized methanol content of 40% to maintain all of the PFAS compounds in solution while maintaining HPLC peak shape. The second approach utilized solid phase extraction (SPE) with ion exchange, mixed mode cartridges, and further cleanup using graphitized carbon. The retention time determined by the optimized reversed phased LC conditions combined with high-resolution mass spectrometry and MS/MS spectra, enabled accurate compound identification across the workflow. On the other hand the sensitivity of the QTRAP System allowed the SPE extract to be injected directly without concentration using nitrogen, which improved recovery of several PFAS compounds, including FOSAs.

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

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Compared with fruits and vegetables, dietary supplements present unique challenges for the detection and quantitation of pesticide residues. The major difficulty is increased levels of non-target substances which interfere and suppress the signal of the target analytes. Even when using highly sensitive and specific techniques such as LC-MS-MS with triple quadrupole mass spectrometry, some false positive signals may be observed from non-target matrix components. Even worse, any substances which are not included in the targeted MRM settings will not be detected.
To overcome these limitations we used a high-sensitivity, high speed quadrupole-time-of-flight mass spectrometer. Using the higher sensitivity and selectivity of this instrument, we developed an LC-MS analysis method using scan and data-dependent MS-MS to detect pesticides in popular dietary supplements. The tandem mass spectra acquired, made possible by the high scan speed, were used to confirm detections.
Representative samples of botanicals were homogenized by grinding and extracted with acetonitrile accompanied by shaking and sonication. Depending on the complexity of the matrix, samples were additionally cleaned up using dispersive solid phase extraction to remove unwanted matrix components. Using our newly developed method, we are able to characterize the extent of residual pesticides present in popular botanicals with unprecedented sensitivity and

selectivity.

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

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

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

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

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

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

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Acrylamide is a well-known carcinogenic contaminant formed at high temperatures during the cooking of starch containing foods. Acrylamide hit the headlines again internationally in 2018, when a judge in California ruled acrylamide fell under the State's Proposition 65 labeling requirements and EU Regulation 2017/2158 was enacted establishing mitigation measures and benchmark levels for the reduction of the presence of acrylamide in food in Europe. The analysis of acrylamide in processed foods has several analytical challenges to consider, including sufficient retention, the complexity of the matrices and the wide range concentrations likely.
A new method, using modified QuEChERS and LC-MS/MS with a high strength silica C18 analytical column, has been developed, to provide a rapid, cost-effective approach for quantifying acrylamide in coffee. Solid phase extraction (SPE) and dispersive solid phase extraction (dSPE) devices were evaluated, to identify simple and efficient cleanup of the samples, to provide selective MRM transitions.
Single laboratory method validation was completed by spiking known amounts of acrylamide into a selection of store purchased coffee products. Acrylamide-d3 was used as an internal standard to correct for any variability through the whole method, including any LC-MS/MS matrix effects. Validation of the method demonstrated excellent performance in terms of linearity, accuracy, precision and repeatability, in accordance with the criteria outlined in Commission Regulation (EU) 2017/2158. Furthermore, results from the analysis of a coffee reference material demonstrated that the analytical method, using a simple and rapid clean up procedure, was suitable for the determination of acrylamide, in accordance with regulatory requirements.

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

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Functional foods require a safe supply of ingredients with verified authenticity. Analysis of coffee products claiming to be a natural solution to erectile dysfunction and stating to be derived from herbs including Tonkat Ali, have been found to contain synthetic PDE-5 inhibitors. The presence of undeclared ingredients can cause risks to health of consumers. There is a need for cost effective monitoring of adulterants in such functional foods.
Here we describe a rapid initial screen using direct analysis in real time (DART) coupled to ACQUITY QDa Mass Detector and confirmation using LC-HRMS. Samples of coffee were analyzed directly, without any sample preparation, using the DART-MS. Ions corresponding to the protonated molecular ions of caffeine, sildenafil and tadalafil at m/z 195, 475 and 390 respectively, were observed in the mass spectra obtained. Analysis of a second coffee sample, generated mass spectra with ions consistent with other PDE-5 inhibitors previously reported in the literature. The same coffee samples were extracted with solvent to perform further studies using untargeted analysis using LC-HRMS. Caffeine, sildenafil and tadalafil were all detected in the extracts by searching the data for entries in a predefined list created from a scientific library and the identify confirmed using accurate mass measurement and isotope patterns of molecular species and fragment ions. Tentative assignments of other suspected PDE-5 inhibitors were made using the Discovery tools available in UNIFI. Isolation of these candidates and further characterization will allow for their definitive identification.

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

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

world, require (sub) parts per trillion (ppt) detection of PFAS in various types of environmental samples. An approach based upon direct injection of water samples was developed for the determination of a wide range of legacy and emerging (e.g. GenX, ADONA, F53-B) PFAS compounds. This approach utilizes little sample preparation and requires a highly sensitive mass spectrometer for detection. Samples of water had been spiked with a mixture of PFAS compounds prior to being received for analysis. The performance of the method was assessed and passed all the required QC criteria including linearity (R2 values > 0.99) and trueness (70 -130%). Detection limits were established to be in the low ppt range. By simplifying the sample preparation step, sample throughput can be drastically increased as well as reducing chances for sample contamination from inherent PFAS in typical laboratory supplies. The performance and scope of the method makes it a suitable approach for the testing of water samples for a wide range of PFAS, legacy and emerging, at relevant concentrations.

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

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Perfluoroalkyl substances (PFAS) are common persistent environmental contaminants used in the production of many consumer products. They are used as surfactants and for nonstick, stain, and water resistance coatings and in firefighting foams. Global use of these compounds over decades has led to their release into the environment. PFAS are classified as persistent organic pollutants.
Currently, there are no regulations pertaining to PFAS in water in the USA, although PFOS and PFOA are included in many drinking water health advisories (e.g. 70 ppt [ng/L]). In Europe, the Water Framework Directive and Drinking Water Directive have set minimum quality standards of PFOS and PFOA, which range from the ppb to sub-ppt levels. Such regulations have driven the need for highly sensitive analytical measurements to detect PFAS.
Sample preparation, such as described in the ISO 25101 method, is typically applied for enrichment of PFAS in water samples. The scope of the ISO 25101 procedure has been expanded to cover approximately 40 legacy and emerging PFAS compounds, including GenX, using a weak anion exchange SPE cartridge. The method was assessed using surface, ground, influent and effluent water. The modified method was found to be robust in all types of matrices tested, with detection limits in the low to sub ppt range, making this method suitable for testing compliance with the guidelines/limits set in both the USA and EU. Recoveries were within the prescribed range of 70 - 130 % and method repeatability was assessed with RSDs < 15%.

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

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Glyphosate continues to cause controversy and so analysis is of considerable interest to governments, the food industry and contract testing laboratories. Many wish to move away from methodology that employs derivatization to save time and expand the scope to cover other polar and ionic pesticides.
Chromatographic retention and separation was optimized using a novel hydrophilic interaction liquid chromatography (HILIC) column, applying an acidified mobile phase gradient, with and without ammonium formate. The performance of a buffered and un-buffered version of the method was compared. Removal of the ammonium formate from the mobile phase resulted in improved sensitivity without compromising chromatographic performance. The aim was to achieve chromatographic retention and baseline separation of isobaric compounds whilst providing maximum sensitivity of all target analytes.
Foods of plant origin were prepared using a modified version of the Quick Polar Pesticides (QuPPe) extraction procedure and spiked with a panel of representative anionic polar pesticides for analysis. All analytes were sufficiently retained detected at concentrations <0.01 mg/kg in matrix-matched standards using the new acidified method. All isobaric pairs (AMPA/fosetyl al and fosetyl al/phosphonic acid) were well separated. The performance of the LC-MS/MS method was assessed using the relevant criteria defined in the SANTE guidance document (SANTE/11813/2017). Linearity was assessed through matrix-matched calibration over a suitable

concentration range (0.001-0.1 mg/kg). Ion ratios and retention times agreed well with reference values and all were within the required tolerances (±30 % and ±0.1 minutes, respectively). The details of a new improved LC-MS/MS method will be presented.

P-70 Determination of Pesticides in Edible Oils

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Detection of pesticides in olive oil has many challenges due to the complexity of the matrix which is high in lipids. Sample cleanup is, therefore, crucial to ensure robust methodology. Traditional method involved liquid-liquid extraction followed by gel permeation chromatography (GPC) cleanup. This technique is undesirable due to high solvent consumption and lengthy extraction and cleanup times. This poster presents an alternative, extraction and cleanup procedure optimized for the simple and reliable determination of multi-residue pesticides in edible oils by GC-MS/MS.
Edible oils were homogenized and spiked with known concentrations of pesticides. The extraction was optimized, followed by a simple pass through cleanup. All extracts were run on GC-EI-MS/MS, in MRM mode, using a splitless injection of 1 µL. Validation was completed in accordance with European guidelines (SANTE/11813/2017) evaluating sensitivity, selectivity, repeatability, accuracy and identification criteria.
The extraction optimized in this study yielded improved method recovery for representative pesticides, when compared to the traditional hexane and acetonitrile extraction procedures. The impact of the pass through cleanup step was also evaluated separately, where analyte recoveries were >70% for all analytes. An alternative type of calibration, namely procedural standards, was employed. This mode of calibration compensates for low extraction efficiency and matrix effects and showed excellent improvements in terms of accuracy and repeatability, where the method's trueness ranged from 99.2 to 108.5 % for a selection of challenging organochlorine pesticides.
Excellent linearity, over the calibration range of 0.005 to 0.1 mg/kg, was achieved for all pesticides with coefficients of determination > 0.995 with residuals <20%. The method's accuracy, repeatability and bias was evaluated from the analysis of spikes at 0.01 mg/kg (n=5), 0.02 mg/kg (n=5) and 0.1 mg/kg (n=5).

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

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

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

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Pesticide residue analysis on fruit and vegetables is essential for the protection of human health and to guarantee international trade. The great number of pesticide residues and the wide range of concentration encountered means that it is necessary to use powerful analytical tools capable of identifying and quantifying a high number of residues at trace levels. Nowadays, the analysis of pesticides is dominated by triple quadrupole (QqQ) technology. However, the recent advances of high-resolution-accurate-mass-spectrometry (HRAMS) instruments make its performance in quantitative analysis fully adequate. This study shows the feasibility of GC-TOF-MS using large volume injection for the multiresidue analysis of 214 pesticides in QuEChERS extracts of tomato and orange. A high degree of sensitivity was achieved with an instrumental limit of identification (LOI) of 2 µg kg-1 for the 86 % of the compounds in tomato and 75% in orange matrix. The instrument presented very good linearity over the concentration range 5-100 µg kg-1 (r2>0.99). Robustness of analytical conditions was measured thorough 40 consecutive injections in orange matrix showing good response, with the intensity of the signal not affected in 75 % of the pesticides. Very good mass accuracy was observed with values < 2 ppm for most pesticides in tomato and orange. The instrument showed great capabilities for the identification and quantification for pesticide residues in fruit and vegetables in agreement with EU AQC procedures. In addition, the high selectivity provided by the instrument allowed the detection of false positives and negatives identified by QqQ.

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

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

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

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

multiresidue method is not necessary, in addition, the absence of water and the low flow that reaches the source increase the sampling efficiency. This research focuses on those compounds whose isomers have a different toxicological nature like Lambda-Cyhalothrin. Lambda-Cyhalothrin is a mixture 1:1 of lambda and gamma isomers. The acute reference dose (ARfD) of gamma isomer is twice that of lambda. Lambda-Cyhalothrin was applied in lettuces under greenhouse conditions. Collections were made at different stages and analyzed to identify if there is any change in the isomers proportion. A validation study of lambda and gamma cyhalothrin was performed in terms of linearity, matrix effect and precision. The limit of quantification (LOQ) was 5 µg/kg-1 in the different matrices studied. In conclusion, the results obtained in the validation showed that SFC-MS/MS facilitates the enantioseparation of pesticides within a short period of time and obtaining very good LOQs of each isomer.

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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Neonicotinoids are a relatively new class of insecticides extensively used in seed and soil treatment. They act as nicotinic acetylcholine receptor (nAChR) agonists, which affect the central nervous systems of insects resulting in paralysis and death. They can be taken up through the roots of plants and translocated to their leaves, flowers, and pollen. Up to date, it is still challenging to have a comprehensive LC-MS workflow for the simultaneous measurements of imidacloprid, thiamethoxam, clothianidin, and imidacloprid metabolites (e.g., OH-imidacloprid and imidacloprid olefin).
A generic QuEChERS extraction was applied for pollen and plant material. Samples were spiked with labeled internal standards, extracted with acetonitrile, and salted out with MgSO4 and NaCl. The mixture was centrifuged, and 1 ml of supernatant was subjected to dispersive solid-phase extraction (dSPE) clean-up. Analysis of neonicotinoids and metabolites was performed on a UHPLC with tandem triple quadrupole (LC-MS/MS). LC separation was carried out on a C18 column (100 × 2.1 mm, 2.6 µm) with injection volume of 2 µl. The mobile phases consisted of A: water:methanol (98:2 v/v) with 5mM ammonium formate and 0.1% formic acid and B: methanol:water (98:2 v/v) with 5mM ammonium formate and 0.1% formic acid.
Results showed good linearity over the concentration range for neonicotinoids and metabolites and correlation coefficients were at r2 > 0.990. Method detection limits (MDLs) ranged from 0.05 – 0.3 ng/g in plant tissue, and from 0.1 – 1 ng/g in pollen. The intra- and inter-day precision (%RSD) were all within 15% of the reference values, and the accuracy ranged from 78 to 110%. Application of dSPE for sample cleanup indicated minor matrix effects when comparing the peak response of analytes in post-spiked extracts with the peak response of analytes in pure solvent. The extraction recoveries ranged from 85 to 101% for target analytes in plant tissues and pollen.

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

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Veterinary drugs are broadly defined as chemicals that are used to protect animals from contracting disease, promote growth, and in some cases provide aesthetic qualities in food production. The inappropriate use of veterinary drugs can have adverse effects on animals, the environment, and human health.
The determination and efficient analysis of veterinary drugs is an important part of routine food quality control. The European Union (EU) and others have developed specific regulations to address these growing concerns. The requirements of low limits of quantification in diverse matrices, along with a wide variety of chemical classes and properties of veterinary drugs pose significant analytical challenges. Several methodologies exist which are typically limited in scope to specific chemical classes, labor intensive, and require extensive sample preparation and clean-up.
This study applies a multi-residue, multi-class workflow using a simple modified QuEChERS sample preparation procedure to the analysis of over 160 veterinary drugs by liquid chromatography-triple stage mass spectrometry (LC-MS/MS). The workflow will be evaluated using a certified reference material known as BOTS-1. It contains incurred veterinary drug residues in bovine muscle. The certified values are based on results from the National Research Council Canada (NRC), the Canadian Food Inspection Agency (CFIA), the USDA, and the German Federal Office of Consumer Protection and Food Safety (BVL) using tandem LC-MS/MS. The sample preparation method and analysis of the BOTS-1 sample will be described, along with performance data obtained vs. the certified values.

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

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Laboratories are constantly challenged to analyze more classes of pesticides at lower concentrations in several different commodities, with faster turnaround times and little if any increase in costs. They will also expect that residues will not go undetected and all results can be verified by associated analytical quality control data compliant with specific method performance guideline criteria.
One of the most challenging groups of pesticides are the polar anionic pesticides, such as glyphosate, perchlorate, chlorate and the like, which often occur as residues in food, but are not always included in pesticide monitoring programs. A number of analytical approaches were considered, including HILIC, and non-suppressed IC, but IC with eluent suppression offered a number of benefits. This presentation will therefore focus on the development of a new, validated IC-MS/MS based workflow for the robust, sensitive and reliable determination of 15 polar anionic pesticides and metabolites at low µg/kg levels in a single run. The development was not straightforward so the presentation will highlight the challenges encountered and steps taken to successfully overcome these.
The workflow uses a modified Quick Polar Pesticide (QuPPe) extraction with cartridge solid phase extraction clean-up and has been thoroughly tested and validated. Results for wheat and leek matrices are compliant with SANTE guidelines, and EU MRLs. Quantification limits are 10ug/kg with % RSDs typically <10 %. Recoveries with and without internal standards and using matrix-matched calibration, and procedural matrix extracted calibrations will be presented.

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

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Polar pesticides include some of the most frequently used pesticides such as glyphosate and glufosinate. Due to the complexity of food matrices and the high polarity of anionic pesticides, their simultaneous and sensitive analysis is challenging. This presentation provides information on the development and validation of a new integrated 'sample-to-results' workflow for the reliable and sensitive quantitation of polar anionic pesticides and contaminants in food. The workflow is based on the use of high capacity ion exchange columns with post column eluent suppression coupled to a high sensitivity triple quadrupole mass spectrometer (IC-MS/MS).
The workflow is based on a modified version of QuPPe (Quick Polar Pesticides) extraction procedure. In QuPPe method, the absence of a liquid partitioning step and/or solid phase clean-up step, results in 'dirty extracts' containing high concentrations of matrix co-extractives, thus the separation and accurate quantification of analytes in QuPPe extracts is difficult. Analysts attempt to mitigate these issues by the use of labelled internal standards and various SPE cartridges.
The results demonstrate that the workflow based on IC-MS/MS with clean-up step can overcome many of the issues experienced with previous methods reported for the analysis of polar pesticides. the IC-MS/MS workflow approach enables aggregation of separate methods into a single analysis improving productivity, while the high capacity ion exchange columns can withstand higher sample loading enabling the analysis of lower concentrations of polar analytes in the more difficult, but relevant matrices, such as cereals and cereal products.

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

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A large number of pesticide residues is used worldwide in food products for preventing, destroying or controlling pest activity. Therefore, for consumer protection, regulatory agencies have established maximum residue levels (MRLs). For baby food products MRLs are even lower compared to other food commodities, and therefore

sensitive analytical methods that allow simultaneous analysis of a large number of pesticides in challenging matrices are required. We have developed a 15-min multiresidue method for quantitation and screening of pesticide in baby food using a triple quadrupole mass spectrometer, coupled to a HPLC in a single run with polarity switching.
Baby food was obtained from a local retail store. Samples were extracted using a QuEChERS extraction kit. Briefly 10g of sample was weighed and 10ml of ACN was added. The mixture was shaken followed by addition of salts from a pre-prepared QuEChERs extraction mix and then shaken again. Followed centrifugation the supernatant was collected. Pesticide standards were spiked into the matrix extracts at different concentration levels ranging from 0.05 to 200 ppb. Chromatographic separation was performed on a Vanquish UHPLC system using an Accucore aQ column. Mass spectrometric analysis was performed on a TSQ Quantis triple quadrupole mass spectrometer with polarity switching. Data analysis was performed with Trace Finder software.
We have developed a multiresidue LC-MS/MS method for the analysis of pesticides in baby food using a triple quadrupole mass spectrometer. The 15-min method allows for pesticide quantitation and screening at low concentration levels (ppb) which are required for baby food. Pesticide confirmation was performed based on one or two ion ratios. Optimum SRM transitions (quantifier and qualifier ions) were determined for each compound by optimizing RF lens values and collision energies for each of the 250 pesticides in neat standards. Preliminary data obtained from matrix-matched standards indicates that the performance of the method in terms of RT reproducibility, detection limits, CVs, RSDs, accuracy is excellent. Limit of detection and limit of quantitation were between 0.5 ppb to 5 ppb for all pesticides tested. Future work will focus on method validation according to the SANTE guidelines. We will discuss in details the data, results and method validation.

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

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Pesticides are routinely applied to crops for preventing, destroying or controlling pest activity. Given the large number of pesticides used and the globalization of the food industry, multiresidue methods offer a great advantage allowing analysis of hundreds of pesticides in a single run. We have implemented a multiresidue method for the analysis of 250 pesticides on an Orbitrap ID-X Tribrid mass spectrometer utilizing an automatic background extraction workflow (AcquireX).
Strawberry samples were obtained from a local retail store. Following homogenization, strawberry samples were extracted using a QuEChERS extraction kit. The matrix extracts were spiked with the pesticide standards (250 pesticides) at different concentration levels ranging from 0.05 to 200 ppb. Chromatographic separation was performed on a Vanquish UHPLC system using an Accucore aQ column. Mass spectrometric analysis was performed on an Orbitrap ID-X Tribrid mass spectrometer using AcquireX workflow, for automated generation of background exclusion list, or data dependent acquisition (DDA).
We have evaluated the performance of a multi-residue pesticide method utilizing high mass accuracy and high resolution for semi-quantitation and screening of pesticide residues in a strawberry matrix. Excellent detection limits, reproducibility, linearity and accuracies were obtained. Overall, for 250 pesticides, out of 251 tested, the LODs were at/or below 5 ppb with 215 pesticides having LODs at/or below 1 ppb. LOQs were below 5 ppb for 247 pesticides tested. When the AcquireX workflow was applied for automated background exclusion we observed a significant increase in the number of library matches compared to DDA, especially at the lower concentration levels. For instance, at a spiked concentration of 0.5 ppb the presence of 19 pesticides was confirmed via library search with DDA. When utilizing the AcquireX workflow, at the same concentration level, the presence of 145 pesticides was confirmed. Similar trends were observed at a concentration level of 1 ppb in which we observed 178 library matches with AcquireX versus 65 library matches with DDA.

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

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

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

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

P-89 Extraction and Cleanup of Acrylamide in Complex Matrices Using Accelerated Solvent Extraction

Followed by Liquid Chromatography Tandem Mass Spectrometry (LC-MS/MS)

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Acrylamide is formed during the cooking process of certain plant-based foods which are rich in carbohydrates and low in protein. Specifically, it forms when asparagine reacts with sugars such as glucose at high temperatures. Acrylamide was detected in fried foods by the Swedish National Food Authority in 2002. Since then, many food laboratories have successfully performed determinations for this compound on a variety of different food matrices. Acrylamide is a known carcinogen in animals.
Accelerated solvent extraction is an excellent technique for extraction of acrylamide from various fried food products; until recently, however, extraction of this compound from matrices such as coffee and chocolate has proven difficult. Traditional extraction techniques are time consuming and may cause bottlenecks in sample preparation. This study compares accelerated solvent extraction methods versus manual extraction procedures. This method combines the extraction of low levels of acrylamide from coffee and chocolate with an in-cell, solid-phase cleanup step. Acrylamide is then quantitated using LC/MS/MS.

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

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Increasing food safety concerns and the growing agricultural trade has resulted in stringent pesticide regulations globally. To comply with such regulatory standards, screening methods for large numbers of pesticides is becoming important. Using liquid chromatography coupled Tandem quadrupole mass spectrometry offers highly sensitive, specific and selective detection in complex matrices for analysis of multi-class pesticides in food samples (wine and olive oil).
An Accucore aQ column was utilized for the separation of all analytes within 15minutes. Wine and olive oil were extracted with organic solvent using a simplified QuEChERS method and lipid removal cartridge to help remove excess fat or oil from olive oil and 1uL of sample was injected with a Vanquish Flex HPLC coupled to a TSQ Quantis triple quadrupole mass spectrometer. A multi-residue method was developed for screening (550+) and quantitation of approximately 300 pesticides in one 15-minute run with polarity switching. Ion ratios were used to confirm each analyte (±30%), plus accuracy of retention time to ±0.1min to show robustness of the method which are required for the EU SANTE Guidance 11813_2017. All pesticides analyzed show excellent Limits of Quantitation and Detection between 0.5 to 10ppb, while reproducibility (injection = 8/level) showed excellent precision and linearity with R2=0.9900. Utilization of the lipid removal cartridge showed good %Rec between 10ppb and 50ppb between 70-120% which is within the SANTE Guidance. Unknown samples of wine and olive oil were also screened. Furthermore, the method was developed using software with built-in workflows for streamlining method development and routine analysis.

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

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The QuEChERS method has been shown to be practical for pesticide analysis on a number of different sample types and is increasingly being employed on more difficult matrices. Unfortunately some matrices either by their nature or their economic value like cannabis can be difficult to analyze with just the QuEChERS method

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

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

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

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

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

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

Capillary GC Column

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Paprika is a spice made from the drying of sweet peppers, and it is used for flavor and color in many types of cuisine. Contamination with polynuclear aromatic hydrocarbons (PAHs) can occur from environmental exposures to the pepper plants and/or during the drying process. The use of herbs in cooking and food production has become increasingly popular, thus exposure to PAHs, specifically those with carcinogenic properties, is of concern.
The sample preparation methods used in the testing of PAHs in herbs and spices require solvent extraction, followed by a cleanup step. The traditional cleanup methods that have been used include gel permeation chromatography (GPC), and solid phase extraction (SPE) with silica gel. A new approach for cleanup of high background samples to be analyzed for PAHs is a dual layer SPE cartridge containing Florisil® and Z-Sep/C18. This cartridge, available commercially under the name "EZ-POP NP", has been used for direct extraction and cleanup of edible oil samples analyzed for PAHs. In this work, PAHs were analyzed from paprika using an optimized QuEChERS extraction followed by cleanup with the EZ-POP NP SPE cartridge. Compared to QuEChERS cleanup, the EZ-POP NP yielded a much cleaner extract. Optimization of the extraction and EZ-POP NP cleanup procedures resulted in absolute recoveries of >70% at a spiking level of 10 ng/g. GC/MS/MS conditions were optimized to provide good peak shape and response, especially for the heavier 6 ring PAHs.

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

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

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

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Over the past few years, there has been a trend in the food safety industry towards developing multi-residue screening methods containing 500+ analytes in a single method. The greater the number of analytes in the calibration solution, the higher the probability of having analyte degradation in the calibration solution which

can influence the quality of the data generated. It becomes important to be able to validate the quality of the working calibration solution used for daily calibration. We are developing a method of using internal marker compounds in the working calibration solution to the help validate that the concentration of analytes in the working solution has not degraded. This study shows the progress on how to implement an additional quality control step to increase confidence in data generated from these large multi-component solutions.

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

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The increasing concern around trace-level food and environmental pollutants is driving the demand for more rapid and reliable methods for the identification of chemical residues. Meeting this challenge not only requires the fast deployment of technologies that can differentiate pesticides, PAHs, and other targets from organic interferences, but also requires the complex task of the analyst to search for hundreds of compounds in a wide variety of crop and environmental matrices. This presentation will discuss the application of three different GC/MS platforms for pesticide analysis in a selection of sixteen different brands of strawberries – the number one fruit in the Environment Work Group "Dirty Dozen" list for the last three years.
GC/MSD analysis utilized streamlined data processing with the Agilent's MassHunter SureTarget automated deconvolution and library searching software. GC/MS/MS analysis employed targeting screening for trace level detection with the use of Agilent's Pesticide and Environmental Pollutant MRM Database for over 1100 components. New streamlined screening workflow for GC/Q-TOF is based on a curated accurate mass database of pesticides and environmental contaminants and is designed to comply with SANTE and FDA guidelines. It offers high degree of flexibility of parameters settings for method optimization as well as the efficient data review process.
The benefits of each approach will be discussed in achieving high quality screening with confident quantitative capability.

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

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Despite dramatic improvements in the physical elements of chromatographic and/or mass spectrometric analysis, such as automated sample preparation methodologies or highly robust/performant instrumentation, the rate limiting step for the release of high confidence, high quality results is largely a factor of the methods of data review. This can be due to the highly complex sample matrices involved, along with the requirements to monitor for ever-decreasing levels of environmental contaminants, when acquired on a system under a relatively continuous, high throughput load. Advancements in data processing as well as automated quality assessment can make a significant impact on these practical, real-world factors in the laboratory.
A 'before and after' comparison is provided, from actual laboratory data (e.g. pesticide residue analysis in a cannabis matrix), which illustrates improvements in peak detection, compound identification, and quantitative accuracy/precision based on the use of robust statistical evaluation of the underlying raw data. The numbers and types of manual interventions (e.g. manual peak integration), and their resulting impacts on the results, are described in both discrete terms as well as trended data analytic views. Overall throughput time is also discussed.
Building on these data, potential quality improvement projects are identified, as well as an automated manner in which to assess any such changes in terms of method verification and validation. Method modifications which lower the technical and regulatory burden of data review are highlighted. In particular, method modifications (broadly considered: sample preparation, data acquisition, and/or processing techniques) which lower the frequency and magnitude of manual data intervention are described as a way to generate and release high quality data at a high operational tempo, while maintaining compliance with laboratory SOP and appropriate regulatory requirements.

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

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Polycyclic aromatic hydrocarbons (PAHs) are formed from the incomplete combustion of organic matter. In food, PAHs are generated during the curing and processing of raw foods or meats cooked over an open flame. Trace levels of PAHs are closely monitored in food as diet is the major source of exposure to PAHs. We introduce an optimized sample preparation procedure using Enhanced Matrix Removal – Lipid (EMR) cleanup to remove matrix and lipid interferences for the investigation of low level PAHs in fatty and complex matrix. The challenge of analyzing PAHs by GC/MS/MS is due to the analyte's resistance to chemical reactions. PAHs accumulate rather than degrade and tend to de-sublime making PAHs difficult to vaporize. European Union-regulated PAHs were evaluated using a modified Gas Chromatograph Triple Quadrupole Mass spectrometry (GC/MS/MS) in Electron Ionization mode. The source to be kept clean from PAH and sample deposition by introducing a low flow of hydrogen during analysis. Post-run mid-column backflush provides a longer column lifetime by eliminating the need to bake out the column and less mass spectrometry maintenance by removing high-boiling matrix contaminants.

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

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

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

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Pesticides are used globally as a method to eradicate unwanted pests to enhance crop yields and allow the more frequent re-use of land for crop growth. There are several classes of pesticides which all target different organisms: herbicides (plants), insecticides (insects), acaricides (mites/ticks) and fungicides (fungi). The use of

pesticides is tightly regulated due to their potential toxicity. Pesticides can be harmful to humans if present in concentrations exceeding safe exposure limits. These limits (known as maximum residue limits – MRLs) have been put in place by the World Health Organisation (WHO), the Food and Agriculture Organisation of the United Nations (FAO) and the European Food Safety Authority to ensure the safety of consumers worldwide. It is then the responsibility of individual countries to perform testing of agricultural samples to confirm pesticide residue levels are below the recommended thresholds. This testing if often performed using (U)HPLC coupled to tandem mass spectrometry to allow trace level determination.
This work presents an LC-MS/MS approach for the analysis of a 300 pesticide panel containing multiple classes of pesticides commonly found in foodstuffs and animal feed. A solid-core stationary phase with a unique and wide pH range stable C18 bonding chemistry was used for this multi-residue low level determination method.

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

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

P-103 Contamination of turmeric with lead-containing pigments

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

therefore value of an otherwise inferior turmeric product.

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

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