53RD ANNUAL NACRW

NORTH AMERICAN CHEMICAL RESIDUE WORKSHOP

TradeWinds Island Grand Resort
St. Pete Beach, Florida
July 17-20, 2016

"Bringing Scientists Together to Develop and Validate Better Methodologies"

Photo courtesy of Jack Cochran

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FUTURE MEETING DATES

2017 July 23-26
Naples Grande Resort

2018 July 22-25
Naples Grande Resort

2019 July 21-24
Naples Grande Resort

NACRW
NORTH AMERICAN CHEMICAL RESIDUE WORKSHOP
formerly Florida Pesticide Residue Workshop

July 18, 2016

Dear Attendees, Exhibitors, Sponsors and Guests;

Welcome everyone to the 53rd, NACRW workshop. We extend a warm greeting to our long-time attendees, our international guests as well as our first time participants. We hope everyone is excited to be a part of the workshop presenting great science, networking and fun!

It's my pleasure to highlight some of the events we have planned for you. The 2016 NACRW Organizing and Program Committees have worked very hard to create and offer the best technical program starting with the short course. The short course, *Quantitative Analysis by Mass Spectroscopy: Development and Validation of Methods for Small Molecule Analysis by LC/MS and GC/MS,* will cover a broad range of topics. We are very honored to have two wonderful instructors in Bob Bethem and Mike Filigenzi, their wealth of experience will be extremely valuable to the class participants. Our welcoming reception on Sunday evening will take place on site in the Pavilion exhibit area. This social time is a great opportunity to network with our exhibitors and other workshop participants and is always a great kick-off to the NACRW workshop. Since this will be the last year for the NACRW workshop at the Tradewinds Island Grand before moving to the Naples Grand in 2017 we've planned a wonderful down-home barbeque and glow-in-the-dark volleyball on the beach and more. We hope you will join us. I want to thank all of our workshop sponsors. Without their generosity, we simply would not have an Opening Reception and Monday Evening Social.

This year was the second NACRW Excellence Award sponsored by FLAG Works, Inc. This year the award focuses on Excellence in Detection Relevant to Chemical Residue Analysis. I am pleased to announce awardee André de Kok; recognized for being at the forefront of implementing advanced detection technologies particularly ion trap, single and triple quadrupole, and time of flight for routine enforcement and monitoring for chemical residue analysis. We are honored to have Dr. de Kok at the workshop to address and inspire us.

The technical sessions include a variety of chemical residue related subjects and special interest topics. As the backbone of the workshop, many aspects of residue analysis will be discussed, like pesticide, veterinary drug, difficult residues and matrices. In addition to special topic sessions on toxins, natural products, dietary supplements, cannabis, sample preparation, food fraud and adulteration, emerging contaminants and high resolution mass spectrometry applications. We also have the very informative and popular Updates from the Federal and State Regulatory Laboratories and the Mass Spectrometry Forum.

In addition to our oral sessions please visit the poster session and vendor seminars. The posters authors will be presenting their posters at designated times. This is a great opportunity to engage the authors ask questions and cast your all-important vote for best poster. This is the first year NACRW sponsored by FLAG Works, Inc., offered student poster awards. The students will be attending the workshop and be available to discuss their work during the allotted time. We are pleased to offer Vendor workshops starting on Sunday evening and occur each day of the workshop. This is a great opportunity to hear about the latest developments and discuss your analytical needs with the vendors.

A heartfelt thanks to our exhibitors and sponsors for participating in the workshop. Your generosity and endorsement has allowed us to offer an outstanding workshop, while maintaining affordability for attendees. I want to thank all the wonderful volunteers who are taking time to assist during the workshop and the Organizing Committee, Program Committee, especially Sherry Garris, Brian Eitzer and Executive Director, Teri Besse for all their hard work and commitment. I also want to thank NACRW for this opportunity, it has been a wonderful experience working with everyone.

Now Let's Enjoy!

Joan Stevens, 2016 Organizing Committee President
Sherry Garris and Brian Eitzer, 2016 Program Committee Co-Chairs and the Program Committee members

The George and Wilma Fong Founders Award

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

Past Recipients:

2011 George and Wilma Fong-Founders
2012 Gail Parker
2013 Pat Beckett
2014 Sherry Garris
2015 Jack Cochran

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Garden Courtyard
Pet Play Zone
Sharktooth Tavern
Parking
VENDOR SEMINARS
Palm Court
Island Ballroom
Grand Palm Colonnade CONFERENCE CENTER
South Parking
Jacaranda Hall
Paddleboat Landings
POOL AREA
TAZ Activities
Pirate Island
Tennis Courts
The Pavilion
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Cypress Villa
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EXHIBITS AND POSTERS
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REGISTRATION DESK AND MEETING ROOM
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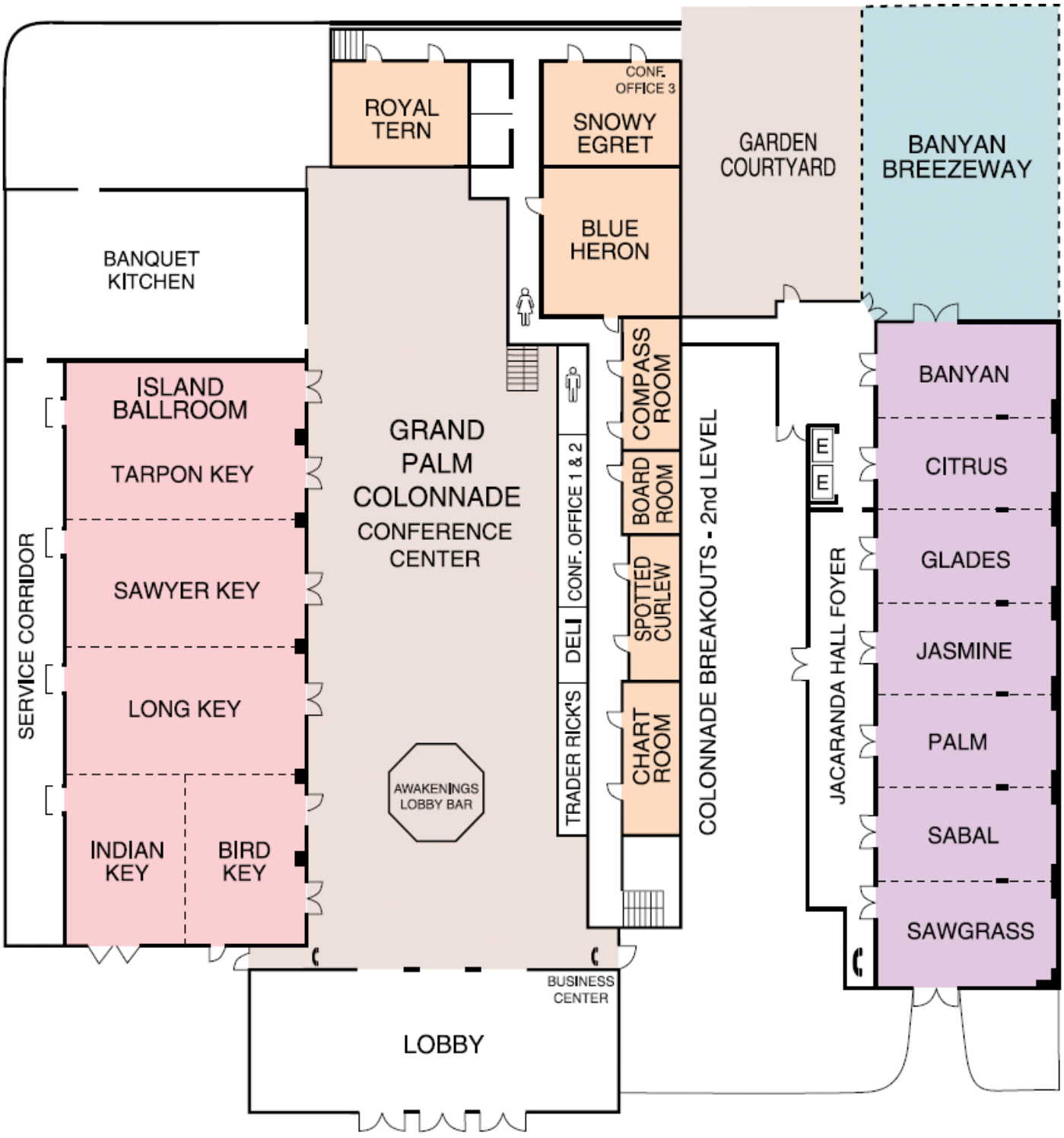

Technical Sessions: Long Key, Bird Key and Indian Key Ballrooms
Exhibits, Posters, Reception: The Pavilion
Vendor Seminars: Sawyer Key/Tarpon Key

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MEETING AT A GLANCE

Saturday, July 16, 2016

8:30 am-4:30 pm	**Short Course: Robert Bethem** and **Mike Filigenzi** *Quantitative Analysis by Mass Spectrometry*	Citrus/Glades

Sunday, July 17, 2016

8:00 am-4:00 pm	**Short Course: Robert Bethem** and **Mike Filigenzi** *Quantitative Analysis by Mass Spectrometry*	Citrus/Glades
1:00 –5:00 pm	Exhibitor Setup	Pavilion
2:00 – 6:00 pm	Registration	Grand Palm Colonnade
4:00 – 5:00 pm	**FDA/State Forum** - government employees only	**Sabal/Sawgrass**
5:15 – 5:45 pm	Moderator and Volunteer Training	Ballrooms
6:15 – 7:15 pm	**Restek Vendor Seminar**	**Tarpon Key**
7:30 – 9:30 pm	**Welcome Reception**	**Pavilion**

Monday, July 18, 2016

All Day	Registration	Grand Palm Colonnade
7:00 – 10:00 am	Poster Board Set Up	Pavilion
7:30 – 8:00 am	Early Morning Coffee	Grand Palm Colonnade
7:00 – 8:00 am	**Waters Corporation Vendor Seminar**	**Tarpon Key**
8:10 – 10:45 am	**Opening Remarks,** **NACRW Excellence Award Presentation and Keynote** **SESSION 1: Advanced Analytical Techniques** **Co-Chairs: Brian Eitzer and Sherry Garris**	Ballrooms
11:00 – noon	**BREAK (Exhibition & Posters)**	**Pavilion**
11:00 – 11:45 am	**Poster Session (authors for odd #s)**	**Pavilion**
12:10 – 1:10 pm	**Thermo Fisher Scientific Vendor Seminar**	**Tarpon Key**
1:15 – 3:00 pm	**SESSION 2: Single/Multi-Pesticide Residue Methods** **Chair: Kelly Dorweiler**	Ballrooms
3:10– 3:55 pm	**BREAK (Exhibition & Posters)**	**Pavilion**
3:10 – 3:40 pm	**Poster Session (authors for even #s)**	**Pavilion**
4:00 – 5:15 pm	**SESSION 3: Vet Drugs** **Chair: Sherri Turnipseed**	Ballrooms
6:00 pm	**Social Event**	

Tuesday July 19, 2016

All Day	Registration	Grand Palm Colonnade
All Day	**Exhibition & Posters**	**Pavilion**
7:15 – 8:15 am	**Early Morning Coffee**	**Pavilion**
7:15 – 8:15 am	**SCIEX Vendor Seminar**	**Tarpon Key**
8:30 – 10:45 am	**SESSION 4: Sample Preparation** **Chair: Jo Marie Cook**	Ballrooms

MEETING AT A GLANCE

11:00 – noon	**BREAK (Exhibition & Posters)**	**Pavilion**
11:00 – 11:45 am	**Poster Session (authors for even #s)**	**Pavilion**
12:10 – 1:10 pm	**Phenomenex Vendor Seminar**	**Tarpon Key**
1:15 – 3:00 pm	**SESSION 5: State/Federal Laboratory Updates** **Co-Chairs: Katherine Carlos and Ping Wan**	Ballrooms
3:10 – 3:55 pm	**BREAK (Exhibition & Posters)**	**Pavilion**
3:10 – 3:40 pm	**Poster Session (authors for odd #s)**	**Pavilion**
4:05 – 5:30 pm	**SESSION 6: Mass Spectrometry Forum** **Co-Chairs: Mark Crosswhite and Amadeo Fernández-Alba**	Ballrooms
5:30– 6:30 pm	**Organizing Committee Meeting**	Ballrooms

Wednesday, July 20, 2016

Until noon	Registration	Grand Palm Colonnade
Until noon	**Exhibition & Posters**	**Pavilion**
6:15 am	Beach Walk/Run	On the Beach
7:45 – 8:15 am	Early Morning Coffee	**Pavilion**
7:15 – 8:15 am	**Millipore Sigma Vendor Seminar**	**Tarpon Key**
8:30 – 10:50 am	**SESSION 7: Natural Toxins/Mycotoxins/Marine Toxins Environmental Contaminants** **Co-Chairs: Marc Engel and Mike Filigenzi**	Ballrooms
11:00 – noon	**BREAK (Exhibition & Posters)**	**Pavilion**
12:00 – 1:00 pm	**Bruker Vendor Seminar**	**Tarpon Key**
1:05 – 2:45 pm	**SESSION 8: Natural Products/Dietary Supplements/Cannabis** **Chair: Alex Krynitsky**	
2:45 – 3:15 pm	**BREAK**	**Grand Palm Colonnade**
2:45 – 3:15 pm	**AOAC Pesticide Contaminant Sub-Committee** **Co-Chairs: Steven Moser and Ping Wan**	
3:15 – 4:55 pm	**SESSION 9: Mass Spectrometry Applications** **Co-Chairs: Jon Wong and Brittany Holmes**	Ballrooms
4:55 – 5:10 pm	**Poster Awards and Closing**	Ballrooms

Thursday, July 21, 2016

User Meetings

7:30-9:30 am	**SCIEX**	**Banyan**
7:30-9:30 am	**Thermo Fisher Scientific**	**Glades**
10:30am-12:30 pm	**Agilent Technologies, Inc.**	**Banyan**

GENERAL INFORMATION

Registration

Check in once at the registration desk at your earliest opportunity
Sunday - 2:00 – 6:00 pm
Monday - 7:00 am – 5:00 pm
Tuesday - 7:30 am – 5:00 pm
Wednesday - 8:00 am – Noon

KEY to Presentation Numbering System

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

Poster Sessions (in The Pavilion, Exhibit Hall)

Hang posters Monday morning from 7:00 am to 10:00 am.
Take down posters between 12 noon to 2:00 pm on Wednesday

Posters may be viewed any time Exhibition is open

Poster Session A (odd#) authors must be at their posters from 11:00 am – 11:45 am on Monday and 3:10 - 3:40 pm on Tuesday
Poster Session B (even#) authors must be at their posters from 3:10 pm – 3:40 pm on Monday and 11:00 - 11:45 am on Tuesday

Poster Prizes

Two poster prizes of $100 each will be awarded this year, and the same poster/author(s) could win both prizes. The People's Choice Poster Award will be determined by popular vote of attendees, and the Judges Choice Poster Award will be determined by the poster committee. The criteria used in each case will be importance of the study, quality of the science, and its presentation (including oral discussion and abstract). Also, UCT will present an award for Excellence in Sample Preparation. **Attendees must place their votes in the ballot box by noon on Wednesday.** ***Get a ticket after you turn in your ballot for the chance to win a door prize.***

Exhibition

Sunday evening reception with light hors d'oeuvres and open bar 7:30 to 9:00 pm and cash bar 9:00 to 9:30 pm
Monday - 11:00 am - 5:00 pm
Tuesday - 7:15 am – 5:00 pm
Wednesday - 7:45 am – noon

Coffee and Breaks

Coffee will be available 7:30 - 8:00 am on Monday morning in the Grand Palm Colonnade and every morning thereafter in the Exhibition Hall (Pavilion). There will also be mid-morning and afternoon refreshment breaks each day. The Monday and Tuesday mid-morning and afternoon breaks, as well as the Wednesday mid-morning break, will be served in the Exhibition Hall (Pavilion). On Wednesday afternoon, the break will be served in the Grand Palm Colonnade. ***All Coffee Breaks are co-sponsored by the Visit St. Pete/Clearwater Convention and Visitors Bureau.***

Announcements

Moderators will make general announcements from the podium. If you need to have an announcement made, fill out an announcement form and submit it to Teri Besse or the onsite audio-visual volunteer. These announcement forms will be available at the registration desk.

Job Placement Bulletin Board

Self-serve message board for those offering or seeking employment or to leave notes for others at the meeting.

Door Prizes

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

Get to Know Your Sponsor

Participate in the "Get to Know Your Sponsor" quiz and win an Apple iPad Pro tablet. A quiz will be provided to you in your registration bag. Simply take the quiz to each sponsor booth, get the right answer and the sponsor will place a sticker on your quiz. After you have completed the quiz, return it to the registration desk no later than Wednesday, July 20th, at 1:30 pm. We will be announcing the winner Wednesday afternoon.

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

You are encouraged to contribute original research and/or review articles to the Journal of Agricultural and Food Chemistry for a special section related to the 2016 NACRW. Please inform Brian Eitzer, 2016 Program Co-Chair (brian.eitzer@ct.gov), by August 31, 2016 if you intend to submit an article. Authors will then be invited by JAFC to submit their manuscripts electronically online through the JAFC website with a deadline of November 15, 2016.

Copies of Presentations

Oral Presentations: Following the meeting, as time and resources permit, oral presentations will be posted on our web site if author permission is granted. There are limitations to what we can post. Absolutely no files will be posted without a speaker's written permission (historically, two thirds of our speakers have given permission). The Power Point files are converted to PDF format, 2 slides per printed page. The file conversion is necessary due to limited server space (the file size of PDF format is roughly 10-20% that of the PPT format). Various security restrictions may be added to the PDF file per speaker's request (such as disabling "copy text" and "print" functions). Some slides containing confidential or proprietary information may be deleted.

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

Meeting Website

www.NACRW.org - the website includes information on current and future NACRW meetings, as well as archives going back to 2005 and copies of the programs from the start of the workshop!

Meeting Evaluations

Look for on-line conference evaluations. Evaluations will be emailed to you daily, so please take a few moments each day to fill them out.

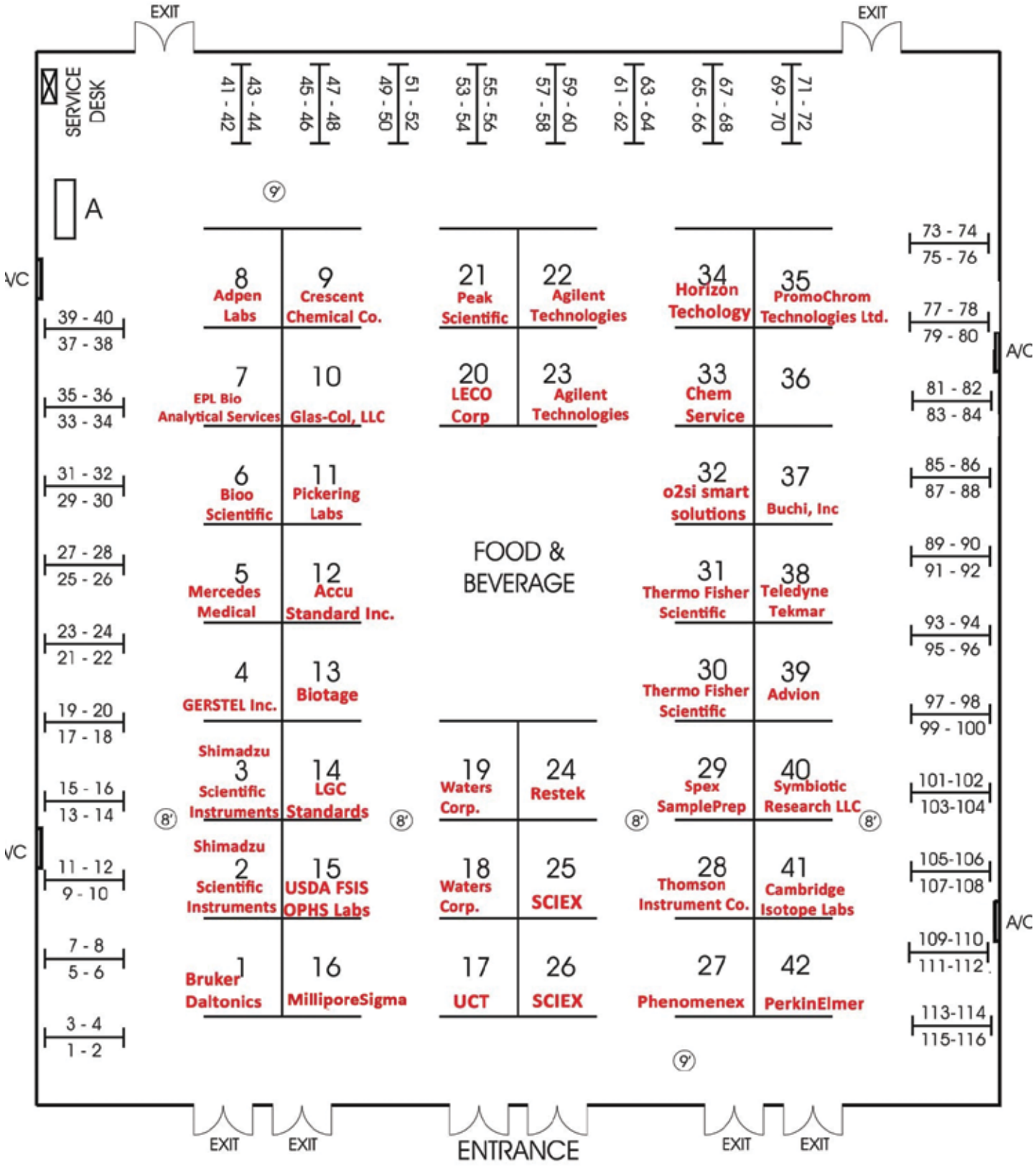
A BIG THANK YOU TO ALL OF OUR VOLUNTEERS, SPONSORS & EXHIBITORS!
The workshop would not be possible without your valuable assistance.

MARK YOUR CALENDAR FOR THE 2017 NACRW

2017 July 23-25 Naples Grande Beach Resort Naples, Florida

Exhibits and Poster Sessions

Location: Pavilion



Exhibitors

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SHORT COURSE

Saturday, July 16, 2016 8:30 am to 4:30 pm
Sunday, July 17, 2016 8:00 am to 4:00 pm
Location: Citrus/Glades

PRE-REGISTRATION IS REQUIRED

"Quantitative Analysis by Mass Spectrometry"

Instructors:

Robert Bethem, (Independent Contractor)

and

Michael Filigenzi, (California Animal Health and Food Safety Laboratory, University of California at Davis)

This introductory/intermediate level two day short course will be focused on the development and validation of methods for small molecule analysis by Liquid and Gas Chromatography-Mass Spectrometry with a particular focus on residue analysis in a regulated environment. The instructors will cover a broad range of topics including: (a) MS instrumentation for quantitative analysis; (b) the general principles of quantitative analysis; (c) instrument optimization; (d) method development; (e) validation; (f) identification and confirmation requirements and (g) method assessment and troubleshooting. Method development and validation discussions will include reviews of US and EU agency requirements as well as issues involved with ISO 17025 accreditation.

This course will provide students all of the basic information required to develop and validate quantitative residue chemistry methods in a regulated environment. Ample time will be provided for general discussion of the topics presented and other current topics in quantitative MS. Prerequisite: Familiarity with the basic principles of mass spectrometry.

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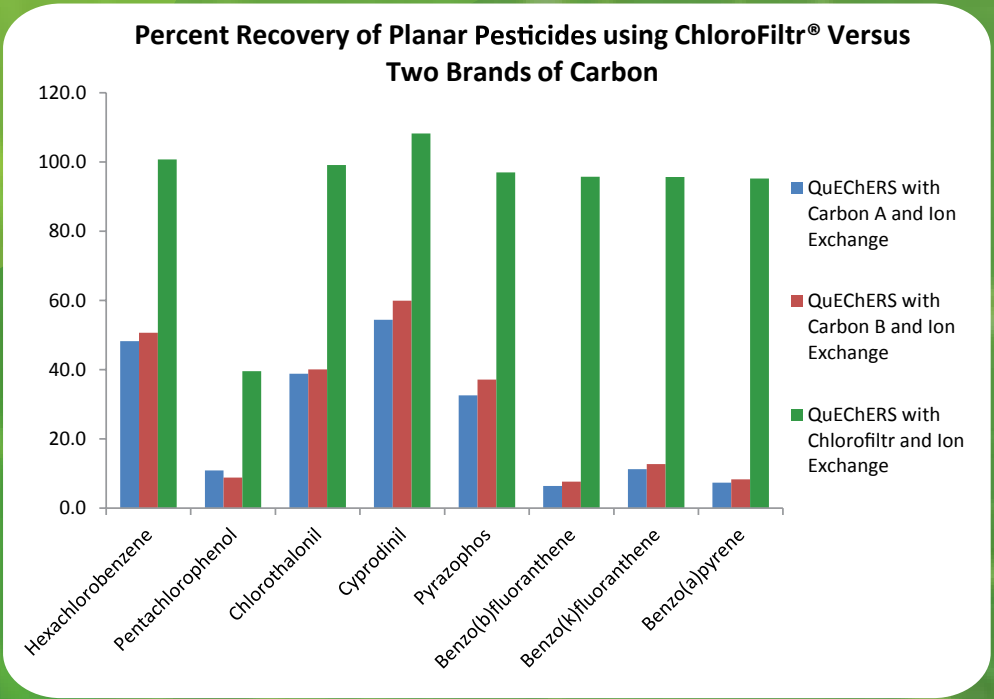

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VENDOR SEMINARS

Food and beverage provided by each company

(PRE-REGISTRATION IS REQUIRED)
Please sign up at the meeting registration desk

V-1 Sunday Evening, July 17, 2016, 6:15 p.m. to 7:15 p.m. RESTEK
Location: Tarpon Key

Cannabis Testing – C'mon, Everybody's Doing It!

Jonathan Keim[1], Julie Kowalski[1], Jeff Dahl[2], Jack Cochran[1],
[1]Restek Corporation, 110 Benner Circle, Bellefonte, PA 16823, USA, [2]Shimadzu Scientific Instruments, Columbia, MD, USA; jonathan.keim@restek.com

Recent years have seen a dramatic increase in public support of cannabis use. With the increase in legal sales of cannabis comes heightened concern regarding consumer safety for cannabis and the plethora of products prepared from it, including edible products. Several states have regulations to control potency of cannabis and are developing meaningful regulations regarding contaminants such as residual solvents, heavy metals and pesticides. It has been difficult to implement the regulatory infrastructure to enforce regulations due to the explosive growth of this market and illegal federal government status. Because of this, an extremely competitive landscape has formed for cannabis testing laboratories, sometimes allowing laboratories employing unscrupulous practices to be more successful than laboratories practicing sound science.

This presentation will introduce you to the cannabis industry as a whole and discuss the most common methods used in the cannabis testing industry, including potency determination, terpene profiling, residual solvents, and pesticide analysis. Special focus will be placed on the analysis of pesticides in cannabis, as this is the most challenging analysis for the cannabis analytical industry.

V-2 Monday, July 18, 2016, 7:00 to 8:00 am WATERS CORPORATION
Location: Tarpon Key

Screening Procedures for Multiclass Contaminants of Pesticides and Mycotoxins in Food Using UPLC-MS/MS and HRMS with Ion Mobility

Gareth Cleland[1] and Simon Hird[2]
[1] Waters Corporation, 34 Maple Street, Milford, MA 01757; gareth_cleland@waters.com
[2] Waters Limited, Stamford Avenue, Altrincham Road, Wilmslow, SK9 4AX, UK; is simon_hird@waters.com

The need for multi analyte screening procedures to efficiently detect violating residues is ever increasing. Routine testing laboratories continue to strive for efficient and reliable sample throughput methodologies, where generic analytical conditions are essential. We will show the development of a screening method for the determination of multiclass multiresidue contaminants in complex foodstuffs, using UPLC coupled with a high sensitivity tandem quadrupole mass spectrometer.

The chemical diversity of mycotoxins and their metabolites produced by plants, microorganism and the animal host can result in a multitude of chemical structures, making screening difficult and prone to errors. Whilst MS techniques are allowing precise determination of the distribution and incidence of some mycotoxins in feeds, these technological advancements become limiting when tracking their metabolites in animal organisms. Furthermore, different levels of toxicity can exist for isomers that cannot be separated by traditional LC-MS/MS methods. We will demonstrate how separation of isomeric species is possible using HRMS coupled with ion mobility.

V-3 Monday, July 18, 2016, 12:10 to 1:10 pm THERMO FISHER SCIENTIFIC

Location: Tarpon Key

Unleashing the Power of QQQ and Orbitrap Technologies on the World of Pesticides

[1] *Prof. Amadeo Fernandez-Alba; European Reference Laboratory for Pesticide Residues in Fruits and Vegetables (EURL-FV), University of Almeria, Spain*
[2] *Charles Yang; Thermo Fisher Scientific, San Jose, CA, US*

Join us to learn how new instrumentation, downloadable methods and software developments help meet your pesticide analysis challenges.

[1]Application of GC-Orbitrap Mass Spectrometry for Pesticide Residues
Gas chromatography with electron ionization and full scan high resolution mass spectrometry with an Orbitrap mass analyzer offers the possibility to perform broad scope analysis with non-targeted acquisitions. This application was evaluated for Pesticide Multi Residue Methods in fruit and vegetables. The applicability for quantitative residue analysis was verified by validation of 150 pesticides at 10 µg/kg in eleven of varying complexity. The pesticides could be identified and quantified according to the EU criteria outlined in SANTE/11945/2015.

[2]Application of LC-Orbitrap Mass Spectrometry for Pesticide Residues
An integrated solution using both GC and LC coupled to HRAM and tandem MS is required for truly comprehensive analysis. The LC-HRAM results showed similar LOQs and excellent discrimination against matrices at R=70k. The HRAM MS/MS spectra acquired in data-dependent mode could be used for identification, and the full scan data were processed retrospectively for screening analysis.
The use of Compound Discover software to detect and identify transformation products, followed by untargeted analysis using HRAM spectral libraries, offers the ability to extend beyond traditional analytical approaches.

V-4 Tuesday, July 19, 2016 7:15 to 8:15 am SCIEX

Location: Tarpon Key

What's in Your Food? New Analytical Techniques, Workflows and Methods to Accurately Detect, Identify and Quantitate Residues and Contaminants with Ease

André Schreiber, Applications Manager Food & Environment Markets, SCIEX, Concord, Ontario, Canada

Food testing is a challenging and complex job. From sample preparation (so many different matrices!) to residue detection (so many different compounds from pesticides to mycotoxins, not to mention the mysterious unknowns!), going from the raw sample to the final result of "What is in this food sample?" is no trivial task. Luckily, a number of powerful analytical tools and workflows are available to ease the pain and help you to answer the question above, quickly and efficiently, but also with the confidence that you arrived at the right result, every time.

In this seminar, we will present data and workflows from QTOF LC-MS/MS instrumentation to demonstrate the power and ease of use for target compound quantitation and identification as well as general unknown screening workflows to detect what you didn't know was present. We will also present our latest results for ultra-trace analysis for glyphosate and its related compounds in complex matrices.
The power and utility of QTOF instruments to provide sensitive quantitative results as well as target compound confirmation with accurate mass and MS/MS spectra will be demonstrated. This provides significantly more information for compound identification than MRM ratios alone and results in less false positive and or negative results. QTOF instruments also provide a powerful tool for detecting and identifying compounds that are not part of the target compound list. The most important feature for these powerful instruments is the software that allows the chemist to analyse, review and report these results. The software workflows for targeted and unknown screening will be presented.

Recently, there has been a large increase in the interest in glyphosate analysis which has created a need to develop more sensitive methodology for glyphosate and its metabolites in a wider variety of matrices. The results of studies to compare various glyphosate methods and the performance of our glyphosate method will be presented.

V-5 Tuesday, July 19, 2016, 12:10 to 1:10 pm **PHENOMENEX**
Location: Tarpon Key

Trace Multiresidue Pesticide Screening in Limited Quantity Cannabis

Scott Krepich and Allen Misa, Phenomenex, Inc., 411 Madrid Avenue, Torrance, CA 90501, USA; ScottK@Phenomenex.com

With the proliferation of legalized medical and recreational marijuana use, multiresidue pesticide testing needs have increased in local testing labs from the dry plant bud samples and edibles. Sensitivity challenges from limited sample quantity availability to high level matrix interferences were overcome by utilizing a very sensitive triple-quadrupole mass spectrometer (Sciex 5500 Triple Quad) and a QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) sample preparation to remove matrix interferences while recovering trace levels of multiresidue pesticides. The chromatography was performed on a Kinetex 5µm Biphenyl HPLC/UHPLC column for selective, efficient, and versatile LC/MS/MS analysis.

V-6 Wednesday, 20, 2016, 7:15 to 8:15 am MILLIPORE SIGMA

Location: Tarpon Key

Analysis of Contaminants in Challenging Samples

Olga I. Shimelis, Katherine K. Stenerson, Michael Ye, Jennifer Claus MilliporeSigma, 595 N. Harrison Rd., Bellefonte, PA 16823, USA; olga.shimelis@sial.com

Lower detection levels and more complex sample matrices present challenges in contaminant testing. In addition to the traditional approaches for extraction and cleanup, new technologies are now available which are more selective and effective in dealing with high background samples. In this seminar, you will learn about two such products. One can be used for the cleanup of highly pigmented samples in pesticides residues analysis, and the other - for cleanup of oily samples in the analysis of non-polar contaminants such as polynuclear aromatic hydrocarbons (PAHs).

For pesticide residue analysis, QuEChERS methodology was applied to extraction of spinach and hops. The cleanup was done using a new sorbent blend containing PSA, zirconia coated/C18 hybrid silica (Z-Sep+), and Envi-Carb™ Y. This sorbent mix, Supel™ QuE Verde, provided sufficient removal of pigmentation while allowing significantly better recovery of planar pesticides over traditional PSA/C18/Carbon mixtures.
In the analysis of contaminants in edible oils, a simple method using a dual layer SPE cartridge was developed and applied to the extraction of PAHs and PCBs. The method produced an extract that is amenable to both GC and HPLC analysis, and is clean enough for analysis by conventional non-MS/MS detection methods such as HPLC/FLD, GC/MS-SIM and GC/ECD.

V-7 Wednesday, July 20, 2016, 12:00 to 1:00 pm BRUKER DALTONICS

Location: Tarpon Key

Mass Spectrometric Strategies for Accurate Screening and Quantitation of Chemical Residues

Dr. André de Kok and Barbara Kiedrowska, NVWA, Wageningen, The Netherlands

With a constantly growing number of potential contaminants and a strong public demand for food and water quality, new stringent regulations are being introduced globally that escalate the need for advanced testing capabilities. Modern systems based on gas and liquid chromatography coupled to mass spectrometry are very well suited to meet the challenges of rapid screening, identification or quantification of trace level chemical residues in complex matrices. This workshop will provide an overview of Bruker's mass spectrometric based solutions and how they provide market leading performance, ruggedness and ease-of-use when used with our innovative application specific content and software. Guest speakers will show real world examples which include the rapid screening of pesticides in food with an ultra-high resolution LC and GC-QTOF systems and the targeted quantification of residues in food and water with both GC-TripleQuad and LC-TripleQuad systems.

2016 NACRW EXCELLENCE AWARD

PRESENTED TO

Dr. André de Kok, NVWA, Wageningen, The Netherlands

Dr. André de Kok is Senior Analytical Chemist at the NVWA – Netherlands Food and Consumer Product Safety Authority. He is the (scientific) coordinator of the National Reference Laboratory (NRL) for Pesticide Residues in Food and Feed, participates in the EURL-NRL network and is responsible for method development, validation and implementation in routine analysis. He received its Ph.D. degree in Analytical Chemistry at the Free University, Amsterdam (NL) in 1983 and has been working in pesticide residue analysis since then. His research interests include QA/QC procedures, extraction methods, sample preparation, pre- and post-column derivatization, chromatographic separation methods and detection techniques, nowadays mainly based on low and high resolution mass spectrometry. He has contributed to 2 book chapters and authored and coauthored over 30 scientific publications and presented over a hundred of lectures and posters at many international symposia and workshops. Dr. André de Kok initiated and was chairman of the first European Pesticide Residue Workshop (EPRW1996), in Alkmaar, The Netherlands and is a member of the EPRW Scientific Organizing Committee and also of the LAPRW (Latin American Pesticide Residue Workshop) Scientific Organizing Committee.

He is a member of the Advisory Group for the 4 EU Reference Laboratories (EURLs) for Pesticide Residues and has contributed since the beginning (1997) to all versions of the well-known EU SANTE "Guidance Document on Analytical Quality Control and Method Validation Procedures for Pesticide Residue Analysis in Food and Feed".

The title of his presentation is: **"The Evolution and importance of Detection Methods in Pesticide Residue Analysis through the years".**

MEETING PROGRAM

Saturday, July 16, 2016

8:30 am-4:30 pm	**Short Course: Robert Bethem** and **Mike Filigenzi** *Quantitative Analysis by Mass Spectrometry*	**Citrus/Glades**

Sunday, July 17, 2016

8:00 am-4:00 pm	**Short Course: Robert Bethem** and **Mike Filigenzi** *Quantitative Analysis by Mass Spectrometry*	**Citrus/Glades**
1:00-5:00 pm	Exhibitor Setup	Pavilion
2:00-6:00 pm	Registration	Grand Palm Colonnade
4:00-5:00 pm	**FDA/State Forum**-government employees only	**Sabal/Sawgrass**
5:15-5:45 pm	Moderator and Volunteer Training	Ballrooms
6:15-7:15 pm	**Restek Evening Seminar** **Cannabis Testing – C'mon, Everybody's Doing It!** Jonathan Keim[1], Julie Kowalski[1,] Jeff Dahl[2], Jack Cochran[1], [1]Restek Corporation, 110 Benner Circle, Bellefonte, PA, USA, [2]Shimadzu Scientific Instruments, Columbia, MD, USA	**Tarpon Key**
7:30-9:30 pm	**Welcome Reception**	**Pavilion**

Monday, July 18, 2016

All Day	Registration	Grand Palm Colonnade
7:00-10:00 am	Poster Board Set Up	Pavilion
7:30-8:00 am	Early Morning Coffee	Grand Palm Colonnade
7:00-8:00 am	**Waters Corporation Breakfast Seminar** **Screening Procedures for Multiclass Contaminants of Pesticides and Mycotoxins in Food Using UPLC-MS/MS and HRMS with Ion Mobility** Gareth Cleland[1] and Simon Hird[2] [1] Waters Corporation, 34 Maple Street, Milford, MA [2] Waters Limited, Stamford Avenue, Altrincham Road, Wilmslow, UK	**Tarpon Key**
8:10-8:20 am	**Opening Remarks** **Sherry Garris**, Chair, FLAG Works, Inc.	**Ballrooms**
8:20-9:25 am	**NACRW Excellence Award Presentation and Keynote Address**	
8:20-8:30 am	**Introduction and Presentation of NACRW Excellence Award** **Joan Stevens, 2016 NACRW President**	
8:30-9:25 am	**Presentation by Excellence Award Winner** **Dr. André de Kok**, NVWA, Wageningen, The Netherlands	
A-1	**The Evolution and Importance of Detection Methods in Pesticide Residue Analysis through the years**	

9:30-10:45 am — **Advanced Analytical Techniques** — **Ballrooms**
Co-Chairs: Brian Eitzer and Sherry Garris

9:30-9:50 am — **Stuart Adams**, FERA Science Limited, Sand Hutton, York, United Kingdom
O-1 — **The Analysis of Polar and Ionic Pesticides by Ion-Exchange Chromatography Tandem Mass Spectrometry; A Tale of Two (and many more) Molecules**

9:55-10:15 am — **Amadeo Fernández-Alba**, European Union Reference Laboratory for Pesticide Residues in Fruit & Vegetables, University of Almeria, Almería, Spain
O-2 — **Evaluation of Simultaneous MS and MS2 Workflows of GC-LC-HRAMS for Analysis of Pesticides in Fruits and Vegetables**

10:20-10:40 am — **Jonathan Byer**, LECO Corporation, Saint Joseph, MI, USA
O-3 — **Targeted Discovery of Disinfection Byproducts in Swimming Pools and Spas**

11:00-noon — **BREAK (Exhibition & Posters)** — **Pavilion**
11:00-11:45 am — **Poster Session (authors for odd #s)** — **Pavilion**

12:10-1:10 pm — **Thermo Fisher Scientific Lunch Seminar** — **Tarpon Key**
Unleashing the Power of QQQ and Orbitrap Technologies on the World of Pesticides
[1] Prof. Amadeo Fernandez-Alba; European Reference Laboratory for Pesticide Residues in Fruits and Vegetables (EURL-FV), University of Almeria, Spain
[2] Charles Yang; Thermo Fisher Scientific, San Jose, CA, US

1:15-3:00 pm — **SESSION 2: Single/Multi-Pesticide Residue Methods** — **Ballrooms**
Chair: Kelly Dorweiler

1:15-1:35 pm — **Del Koch**, EAG/ABC Laboratories, Columbia, MO, USA
O-4 — **Strategies for Analysis of Pyrethroid Insecticides in Complex Matrices**

1:40-2:00 pm — **Agustin Pierri**, Weck Laboratories, Inc., Industry, CA, USA
O-5 — **A Multi-Residue Method for Monitoring Pesticides and Pesticide Metabolites in Groundwater at Low-Levels Using LC-MS/MS with On-Line SPE**

2:05-2:25 pm — **Katerina Mastovska**, Covance Laboratories, Madison, WI, USA
O-6 — **Development and Validation of a Large Multiresidue LC-MS/MS Method using On-Line Dilution and other Useful Features**

2:30-2:50 pm — **Kelli Simon**, US Food and Drug Administration, College Park, MD, USA
O-7 — **Multi-lab Validation Study of 204 Pesticides in Fruits and Vegetables by QuEChERS and LC-MS/MS**

3:10-3:55 pm — **BREAK (Exhibition & Posters)** — **Pavilion**
3:10-3:40 pm — **Poster Session (authors for even #s)** — **Pavilion**

4:00-5:15 pm — **SESSION 3: Vet Drugs** — **Ballrooms**
Chair: Sherri Turnipseed

4:00-4:20 pm — **Hui Zhao**, Covance Laboratories, Madison, WI, USA
O-8 — **Multi-Class, Multi-Residue Veterinary Drug Analysis in Infant Formula and Related Ingredients Using UHPLC-MS/MS**

4:25-4:45 pm **James Wittenberg**, US Food and Drug Administration, College Park, MD, USA
O-9 **Targeted Multiresidue Analysis of Veterinary Drugs in Dairy Products Using Liquid Chromatography-Tandem Mass Spectrometry (LC-MS/MS)**

4:50-5:10 pm **Sherri Turnipseed**, US Food and Drug Administration, Denver, CO, USA
O-10 **Development and Validation of a Screening Method for Drug Residues in Fish and Shrimp using Liquid Chromatography High Resolution Mass Spectrometry**

6:00 pm **Social Event - TradeWinds**

Tuesday July 19, 2016

All Day Registration — Grand Palm Colonnade
All Day **Exhibition & Posters** — **Pavilion**
7:15-8:15 am **Early Morning Coffee** — **Pavilion**

7:15-8:15 am **SCIEX Breakfast Seminar** — **Tarpon Key**
What's in Your Food? New Analytical Techniques, Workflows and Methods to Accurately Detect, Identify and Quantitate Residues and Contaminants with Ease
André Schreiber, Applications Manager Food & Environment Markets, SCIEX, Concord, Ontario, Canada

8:30-10:45 am **SESSION 4: Sample Preparation** — **Ballrooms**
Chair: Jo Marie Cook

8:30-8:50 am **Alexander Krynitsky**, Symbiotic Research, LLC, Budd Lake, NJ, USA
O-11 **Important Considerations with Regard to Sample Preparation when Developing Reliable Analytical Residue Methods**

8:55-9:15 am **Fadi Aldeek**, Florida Department of Agriculture and Consumer Services, Tallahassee, FL, USA
O-12 **Identification of Penicillin G Metabolites under Various Environmental Conditions using UHPLC-MS/MS**

9:20-9:40 am **Tarun Anumol**, Agilent Technologies Inc., Wilmington, DE, USA
O-13 **Comparison of Sample Preparation Techniques and Screening for >120 Veterinary Drugs in Animal Meat**

9:45-10:05 am **Martin Dusek**, Research Institute of Brewing and Malting, Prague, Czech Republic
O-14 **Pesticide Residue Analysis in Hops: Analysts' Nightmare or a Unique Opportunity to Advance**

10:10-10:45 am **Question and Answer Forum**

11:00-noon **BREAK (Exhibition & Posters)** — **Pavilion**
11:00-11:45 am **Poster Session (authors for even #s)** — **Pavilion**

12:10-1:10 pm **Phenomenex Lunch Seminar** — **Tarpon Key**
Trace Multiresidue Pesticide Screening in Limited Quantity Cannabis
Scott Krepich and Allen Misa, Phenomenex, Inc., 411 Madrid Avenue, Torrance, CA, USA

1:15-3:00 pm **SESSION 5: State/Federal Laboratory Updates** **Ballrooms**
Co-Chair: Katherine Carlos and Ping Wan

1:15-1:35 pm **Marc Engel**, Florida Department of Agriculture and Consumer Services, Tallahassee, FL, USA
O-15 **Addressing Consumer Complaints Samples in a Regulatory Laboratory; Food Borne Illness Investigations and Consumer Fraud**

1:40-2:00 pm **Yoko Johnson**, Minnesota Department of Agriculture, St. Paul, MN, USA
O-16 **Establishing the Minimum Analytical Method Validation Procedure for EPA FIFRA Misuse Samples to Meet the ISO/IEC 17025:2005 Requirements**

2:05-2:25 pm **Chris Pappas**, USDA, AMS, S&T, Monitoring Programs Division, Washington, DC, USA
O-17 **The Pesticide Data Program - 25 Years (and counting)**

2:30-2:50 pm **Greg Mercer**, US Food and Drug Administration, Bothell, WA, USA
O-18 **Ongoing Method Development and Harmonization Efforts at FDA Pesticide Monitoring Laboratories**

3:10-3:55 pm **BREAK (Exhibition & Posters)** **Pavilion**
3:10-3:40 pm **Poster Session (authors for odd #s)** **Pavilion**

4:05-5:30 pm **SESSION 6: Mass Spectrometry Forum** **Ballrooms**
Co-Chairs: Mark Crosswhite and Amadeo Fernández-Alba

5:30-6:30 pm **Organization Meeting -** *open to all attendees* Ballrooms

Wednesday, July 20, 2016

Until noon Registration Grand Palm Colonnade
Until noon **Exhibition & Posters** **Pavilion**
6:15 am Beach Walk/Run On the Beach
7:45-8:15 am Early Morning Coffee **Pavilion**

7:15-8:15 am **MilliporeSigma Breakfast Seminar** **Tarpon Key**
Analysis of Contaminants in Challenging Samples
Olga I. Shimelis, Katherine K. Stenerson, Michael Ye, Jennifer Claus
MilliporeSigma, Bellefonte, PA, USA

8:30-10:50 am **SESSION 7: Natural Toxins/Mycotoxins/Marine Toxins and Environmental Contaminants** **Ballrooms**
Co-Chairs: Marc Engel and Mike Filigenzi

8:30-9:15 am **Raphael Kudela**, University of California Santa Cruz, Santa Cruz, CA, USA
O-19 **Freshwater and Marine Algal Toxins: An Old Problem with Expanding Consequences**

9:20-9:45 am **Sara McGrath**, US Food and Drug Administration, College Park, MD, USA
O-20 **Evaluation of Methods for Extraction of Tetrodotoxin and Saxitoxin from Fresh and Salt-Dried Puffer Fish with LC-MS/MS Analysis**

9:50-10:15 am **Simon Hird**, Waters Corporation, Wilmslow, Cheshire, UK
O-21 **Detection of Paralytic Shellfish Toxins and Domoic Acid by LC-MS/MS**

10:20-10:45 am **Robert Trengove**, Murdoch University, Murdoch, Western Australia
O-22 **Pesticide Residues in Human Milk: ppb detection in 1 mL**

11:00-noon **BREAK (Exhibition & Posters)** **Pavilion**

12:00-1:00 pm **Bruker Lunch Seminar** **Tarpon Key**
Mass Spectrometric Strategies for Accurate Screening and Quantitation of Chemical Residues

1:05-2:45 pm **SESSION 8: Natural Products/Dietary Supplements/Cannabis** **Ballrooms**
Chair: Alex Krynitsky

1:05-1:25 pm **Katherine Stenerson**, MilliporeSigma, Bellefonte, PA, USA
O-23 **Contaminant Testing in Marijuana: Pesticides, Mycotoxins and Residual Solvents**

1:30-1:50 pm **Paul Reibach**, Smithers Viscient, Wareham, MA, USA
O-24 **Navigating the Pesticide Related Regulatory Landscape With Respect to Individual State Legal Cannabis Cultivation in the US**

1:55-2:15 pm **Michael S. Young**, Waters Corporation, Milford, MA, USA
O-25 **Pesticide Analysis in Highly Resinous Matrices: Options for Sample Preparation and Cleanup Prior to LC-MS/MS and GC-MS/MS Analysis**

2:20-2:40 pm **Jeff Dahl**, Shimadzu Scientific Instruments, Columbia, MD, USA
O-26 **Poisoned Garden: Pesticides in Cannabis by modified QuEChERS and LC-MS-MS**

2:45-3:15 pm **BREAK** **Grand Palm Colonnade**
2:45-3:15 pm **AOAC Pesticide Contaminant Sub-Committee Meeting** *- open to all attendees* Ballrooms
AOAC Pesticide Subgroup Chairs: Steven C. Moser and Ping Wan

3:15-4:55 pm **SESSION 9: Mass Spectrometry Applications** **Ballrooms**
Co-Chairs: Jon Wong and Brittany Holmes

3:15-3:35 pm **Narong Chamkasem**, US Food and Drug Administration, Atlanta, GA, USA
O-27 **Direct Determination of Glyphosate, Glufosinate, and AMPA in food by Liquid Chromatography/ Tandem Mass Spectrometry**

3:40-4:00 pm **Susanne Ekroth**, Division of Science, National Food Agency, Uppsala, Sweden
O-28 **Swedish Multiresidue Method SweEt goes into Orbitrap Technology**

4:05-4:25 pm **Philip Wylie**, Agilent Technologies, Inc., Wilmington, DE, USA
O-29 **Using Sandwich Injections to add Matrix, Internal Standards and/or Analyte Protectants for the GC/Q-TOF Analysis of Pesticide Residues**

4:30-4:50 pm **Jerry Zweigenbaum**, Agilent Technologies, Inc., Wilmington, DE, USA
O-30 **Profiling maple syrup for Authenticity and Adulteration by Chemical Composition using UHPLC/ QTOF MS**

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

Thursday, July 21, 2016

User Meetings

7:30-9:30 am	SCIEX	Banyan
7:30-9:30 am	Thermo Fisher Scientific	Glades
10:30am-12:30 pm	Agilent Technologies, Inc.	Banyan

MARK YOUR CALENDAR FOR THE 2017 NACRW

2017 July 23-25 Naples Grande Beach Resort Naples, Florida

POSTERS

Session A (ODD NUMBERED POSTERS P1, P3, P5, etc.)
Authors stand by their posters from 11:00 am – 11:45 am on Monday and 3:10 pm - 3:40 pm on Tuesday

Session B (EVEN NUMBERED POSTERS P2, P4, P6, etc.)
Authors stand by their posters from 3:10 pm - 3:40 pm on Monday and 11:00 am - 11:45 am on Tuesday

P-1 **Improved Cleanup for the LC/MS/MS and GC/MS/MS Analysis of Pesticides In Turmeric Powder Using a Novel Dual-Layer SPE Cartridge**
Katherine K. Stenerson, et al.; MilliporeSigma, Bellefonte, PA, USA

P-2 **Planar Pesticide Quantitation from Green Food Matrices Using QuEChERS Cleanup With a Novel Graphitized Carbon Black and Zirconia-based Adsorbent**
William Betz, et al.; MilliporeSigma, Bellefonte, PA , USA

P-3 **Solventless Extraction and Analysis of Pesticides in Foods by Direct Immersion SPME Using an Overcoated Fiber**
Katherine K. Stenerson, et al.; MilliporeSigma, Bellefonte, PA, USA

P-4 **Simultaneous quantitation and confirmation of about 500 pesticide residues in food extracts using LC-QTOF accurate mass spectrometry**
Carsten Baessmann, et al.; Bruker Daltonik GmbH, Bremen, Germany

P-5 **Analysis Ultra-Trace Level 1,2,3-Trichloropropane in Water by GC TQMS System in MRM Mode**
Zicheng Yang and Louis Maljers, Bruker Daltonics Inc, Fremont, CA, USA

P-6 **A Short Screen and Quantitation LC-MS/MS Method for about 250 Positive and Negative Ion Pesticides in a Single Analysis**
Louis Maljers, et al.; Bruker Daltonics Inc, Fremont, CA, USA

P-7 **An Improved Sample Preparation Method for the Analysis of Pesticides in a Challenging Chili Pepper Extract**
Jane E. Guido, Kalsec®, Inc., Kalamazoo, MI, USA

P-8 **Analysis of Pesticide Residues and Mycotoxins in Marijuana using QuEChERS Extraction and ChloroFiltr dSPE Cleanup**
Brian Kinsella, et al.; UCT Inc., Bristol, PA, USA

P-9 **A solid-phase extraction liquid chromatography/tandem mass spectrometry method for analysis of adducts formed between DNA and *epoxide of safrole in vitro and in mice***
Su-Yin Chiang, et al.; China Medical University, Taichung, Taiwan

P-10 **Methodological Advances and Challenges in Long Term Pesticide Monitoring in Lake Erie and Ohio River Basins**
Saptashati Biswas, et al.; Heidelberg University, Tiffin, OH, USA

P-12 **Analysis of 647 pesticides (1,929 MRMs) using a high sensitivity LC-MS/MS with a 10.5 minute gradient**
Jeff Dahl, et al.; Shimadzu Corporation, Manchester, United Kingdom

P-13 **A multi-residue method for the determination of 114 pesticides in cereals and products of animal origin using the QuEChERs extraction and GC-NCI-MS/MS-TQ detection**
Barbara Kiedrowska and André de Kok, NVWA - Netherlands Food and Consumer Product Safety Authority, Wageningen, The Netherlands

P-14 **Performance evaluation of UHPLC-Q-TOF MS and comparison with UHPLC-TQ-MS/MS and Q-Orbitrap MS for pesticide residues analysis in food samples**
André de Kok, et al.; NVWA - Netherlands Food and Consumer Product Safety Authority, Wageningen, The Netherlands

P-15 **Simultaneous Analysis of the Amitraz and its Metabolites in Chili Pepper and Mandarin using LC-MS/MS**
Jeong-Han Kim, et al.; Seoul National University, Seoul, Republic of Korea

P-16 **Simultaneous Analysis of Pesticides and Ochratoxin A in Red pepper powder using LC-MS/MS and GC-MS/MS**
Jongsung Ahn, et al.; National Agricultural Products Quality Management Service, Republic of Korea

P-17 **Survey of Plant Growth Regulators in Consumer Fertilizer Products using LC-MS/MS**
Peter Bradley, PMRA Laboratory Services, Ottawa, ON, Canada

P-18 **Determination of 234 Pesticides in Juices using UHPLC/ESI-MS/MS and QuEChERS**
Wendy Cheung and Jian Wang, Canadian Food Inspection Agency, Calgary Alberta Canada

P-19 **Comparative Validation Study of Microflow-LC and UHPLC with Orbitrap MS for Quantitative Analysis of Pesticides in Food Matrices**
Randy L. Self, et al.; USFDA Pacific Regional Laboratory Northwest, Bothell, WA, USA

P-20 **High resolution/high mass accuracy MS/MS Libraries for Chemical Residues and Contaminants**
Jon W. Wong, et al.; U.S. Food and Drug Administration, Center for Food Safety and Applied Nutrition, College Park, MD, USA

P-21 **Investigation of Free and Reversibly Bound Sulfite using a Non-Targeted High-Resolution Mass Spectrometry Approach**
Katherine S. Carlos, et al.; US FDA, Center for Food Safety and Applied Nutrition, College Park, MD, USA

P-22 **A Simple and Effective Cleanup Procedure to Produce QuEChERS Extracts That Are More Compatible with Gas Chromatography**
Greg E. Mercer, USFDA Pacific Regional Laboratory Northwest, Bothell, WA, USA

P-23 **Determination of Pesticides in High Fat Food Products using a Modification of the Irvine Rapid Analytical Method (IRAM) with LC-MS/MS**
Irene Cassias, et al.; U.S. Food and Drug Administration, Pacific Regional Laboratory Southwest, Irvine, CA, USA

P-24 **A QuEChERS Sample Extraction Procedure Modified with Enhanced Matrix Removal Sorbent for Determination of Pesticide Residues in Edible Oil**
Eugene Chang, et al.; Pacific Regional Laboratory Southwest, U.S. Food and Drug Administration; Irvine, CA, USA

P-25 **Sensitive and Fast Analysis of Glyphosate, Glufosinate and AMPA in Food Matrices with HILIC HPLC and Mass Spectrometry**
Madison Hanson, et al.; Pacific Regional Laboratory Southwest, U.S. Food and Drug Administration, Irvine, CA, USA

P-26 **Differential Mobility Spectrometry for the Analysis of Stilbene Hormone Residues in Seafood**
Wendy Andersen et al.; Animal Drugs Research Center, U.S. Food and Drug Administration, Denver, CO, USA

P-27 **Optimization of a Cleanup and Extraction Procedure for the LC/MS analysis of Veterinary Drugs in Fish and Shrimp**
Joseph Storey, et al.; Animal Drugs Research Center, U.S. Food and Drug Administration, Denver, CO, USA

P-28 **Development and validation of a LC/MS/MS method for the determination of isoeugenol in finfish**
Lin Ye, U.S. Food and Drug Administration, Southeast Regional Laboratory, Atlanta, GA, USA

P-29 **Development and validation of a direct determination of glyphosate, glufosinate, and aminomethylphosphonic acid in honey by liquid chromatography/tandem mass spectrometry**
Narong Chamkasem, Southeast Regional Laboratory, U.S. Food and Drug Administration, Atlanta, GA, USA

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Susan Genualdi, et al.; U.S. Food and Drug Administration, College Park, MD, USA

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Sherri Turnipseed, et al.; Animal Drugs Research Center, US FDA, Denver, CO, USA

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Lance P. Ford, et al.; Bioo Scientific Corp., Austin, TX, USA

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Marc E. Engel, Florida Department of Agriculture and Consumer Services, Tallahassee, FL, USA; and Donald M. Axelrad, Institute of Public Health, Florida A and M University; Tallahassee, FL

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Jo-Marie Cook, Florida Department of Agriculture and Consumer Services, Division of Food Safety, Tallahassee, FL, USA

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Amy N. Brown and Jo Marie Cook, Florida Department of Agriculture and Consumer Services, Chemical Residue Laboratories, Tallahassee, FL, USA

P-36 **Identification of Penicillin G and its Metabolites in Citrus Fruits Affected by Huanglongbing using UHPLC-MS/MS**
Fadi Aldeek, et al.; Florida Department of Agriculture and Consumer Services, Division of Food Safety, Chemical Residue Laboratory, Tallahassee, FL, USA

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Jessica D. Murillo, et al.; Florida Department of Agriculture and Consumer Services, Chemical Residue Laboratories, Tallahassee, FL, USA

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Rebecca Kitlica and Mariana Herceg, Florida Department of Agriculture and Consumer Services, Chemical Residue Laboratories, Tallahassee, FL, USA

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Alex Yiannikouris, et al.; Waters Corporation, Milford, Man USA

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Michael S. Young, et al.; Waters Corporation, Milford, MA, USA

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Joe Romano, et al.; Waters Corporation, Waters Corporation, Milford, MA, USA

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Joe Romano, et al.; Waters Corporation, Waters Corporation, Milford, MA, USA

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Gareth Cleland, et al.; Waters Corporation, Waters Corporation, Milford, MA, USA

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Gareth Cleland, et al.; Waters Corporation, Waters Corporation, Milford, MA, USA

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Simon Hird, et al.; Waters Corporation, Wilmslow, UK

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Simon Hird, et al.; Waters Corporation, Wilmslow, UK

P-47 **Multiresidue Determination of Pesticides in a Fatty Matrix: an Alternative Cleanup for QuEChERS Extracts Prior to GC-MS and LC-MS Analysis**
Michael S. Young, et al.; Waters Corporation, Milford, MA , USA

P-48 **Novel Residue Analysis of Various Food Samples using GCxGC-HRMS with Encoded Frequent Pulsing™**
Scott Pugh et al.; LECO Corporation, St. Joseph, MI, USA

P-49 **Glyphosate analysis in soy beans, corn and sunflower seeds by HPLC with post-column derivatization and fluorescence detection**
Sareeta Nerkar and Maria Ofitserova, Pickering Laboratories, Inc, Mountain View, CA, USA,

P-50 **Analysis of N-Methyl Carbamate Pesticides in Food by HPLC with Post-Column Derivatization and Fluorescence Detection**
Sareeta Nerkar and Maria Ofitserova, Pickering Laboratories, Inc, Mountain View, CA, USA,

P-51 **Extraction of Acrylamide from coffee and potato chips (crisps) using supported liquid extraction (SLE+) prior to LC-MS/MS Analysis**
Kristin Jones, Biotage, Charlotte, NC, USA

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Kristin Jones, Biotage, Charlotte, NC, USA

P-53 **Development of a Cleanup Method Incorporating a Novel SPE Media for the Analysis of Patulin in Apple Juice Using LC-MS/MS**
Adam Senior, et al.; Biotage GB Limited, UK

P-54 **Development of a Multiclass Cleanup Method Incorporating a Novel SPE Media for the Analysis of Mycotoxins in Grain Using LC-MS/MS**
Adam Senior, et al.; Biotage GB Limited, UK

P-55 **Quantitate Analysis of Leachable Contaminates of Bottled Water at Elevated Temperatures**
Alexandria Pavkovich, et al.; Restek Corporation, Bellefonte, PA, USA

P-56 **Accuracy Evaluation of a Free, Web-Based Tool for Gas Chromatographic Modeling for Multi-Class Chemical Contaminants Analysis**
Amanda Rigdon, et al.; Restek Corporation, Bellefonte, PA, USA

P-57 **Shoot-and-Dilute Gas Chromatography-Mass Spectrometry: Polycyclic Aromatic Hydrocarbons Quantification in Tea using Modified QuEChERS Extraction and No Sample Cleanup**
Julie Kowalski, et al.; Restek Corporation, Bellefonte, PA, USA

P-58 **Prolonging GC-MS/MS Performance for Pesticide Analysis: Shoot-and-Dilute Injection and Analyte Protectants**
Julie Kowalski and Jack Cochran, Restek Corporation, Bellefonte, PA, USA

P-59 **Improving the Analysis of Phenylurea Herbicides in Drinking Water and Soft Drinks using Automated Solid Phase Extraction as a Preparation for HPLC-UV Analysis**
Don Haertel, et al.; Horizon Technology, Inc, Salem, NH, USA

P-60 **Enhanced Food Safety Separations using Superficially Porous Particle Column Technology**
Sharon Lupo, et al.; Restek Corporation, Bellefonte, PA, USA

P-61 **Comparison of Sample Cleanup Methods (GPC and EMR-Lipid) for Multiresidue Pesticide Analysis in Avocado by GC/MS/MS**
Kumi Shiota Ozawa, et al.; Agilent Technologies Brasil Ltda

P-62 **Combined contaminant and multi-residue testing by LC/MS/MS in spices and herbal food supplements**
Thomas Glauner, et al.; Agilent Technologies Sales & Services GmbH, Waldbronn, Germany

P-63 **Analysis of Multiple Pesticide Residues in Fruits and Vegetables using GC/Q-TOF and EI Accurate Mass Pesticide Library**
Kai Chen, et al.; Agilent Technologies, Inc., Santa Clara, CA, USA

P-64 **Maintaining Sensitivity and Reproducibility with the Self Cleaning Ion Source for Pesticides in Food and Feed**
Jessica Westland and Elizabeth Almasi, Agilent Technologies, Inc., Wilmington, DE, USA

P-65 **Accurately Identify and Quantify A Hundred Pesticides in a Single GC Run**
Jessica Westland and Tom Doherty, Agilent Technologies, Inc., Wilmington, DE, USA

P-66 **Significant improvement in GCMS screening of pesticides by use of a High-Efficiency Source and spectral deconvolution**
Melissa Churley and Bruce Quimby, Agilent Technologies, Inc., Santa Clara, CA, USA

P-67 **Aflatoxin Analysis in Infant Formula with Enhanced Matrix Removal — Lipid by LC/MS/MS**
Megan Juck, et al.; Agilent Technologies, Inc., Wilmington, DE, USA

P-68 **Determination of Mycotoxins in Peanut with Enhanced Matrix Removal – Lipid and Liquid Chromatography-Tandem Mass Spectrometry**
Megan Juck, et al.; Agilent Technologies, Inc., Wilmington, DE, USA

P-69 **Targeted Veterinary Drugs Screening in Animal Tissues Using Agilent EMR QuEChERS Kit and 6470 Triple Quadrupole Mass Spectrometer**
Dan-Hui Dorothy Yang, et al.; Agilent Technologies, Inc., Santa Clara, CA, USA

P-70 **An Optimal Method for the Analysis of Pesticides in a Variety of Matrices**
Jessica Westland, Agilent Technologies, Inc., Wilmington, DE, USA

P-71 **Two-gram Incurred Food Samples Using Mini-QuEChERS, Cryomilling and GC/MS/MS Analysis with a High Efficiency Ion Source**
Melissa Churley and Joan Stevens, Agilent Technologies, Inc., Santa Clara, CA, USA

P-72 **Improving Pesticides Analysis on GC/MS/MS for Complicated Samples by Increasing Matrix Removal**
Limian Zhao et al.; Agilent Technologies, Wilmington, DE, USA

P-73 **Complete Workflows for Food Pesticide Residue Laboratories Using Tandem and High Resolution Accurate Mass (HRAM) LC/MS Instrumentation**
Ed George et al.; Thermo Fisher Scientific,San Jose, CA, USA

P-74 **Meeting the European Commission Performance Criteria for the Use of Triple Quadrupole GC-MS/MS as a confirmatory Method for PCDD/Fs in Food and Feed Samples**
Richard Law, et al.; Thermo Fisher Scientific, Cheshire, UK

P-75 **An assessment of GC Orbitrap MS technology for the routine screening and quantification of pesticide residues in food**
Paul Silcock, et al.; Thermo Fisher Scientific, Manor Park, Runcorn, UK

P-76 **The analysis of polar ionic pesticides by ion-exchange chromatography tandem mass spectrometry: the possible solution to a longstanding problematic analysis?**
Richard J. Fussell, et al.; Thermo Fisher Scientific, Hemel Hempstead, UK

P-77 **Determination of Pesticide Residues in Drinking Water Using Automated Solid Phase Extraction with GC-NPD**
Sergio Guazzotti and Aaron Kettle, Thermo Fisher Scientific, Sunnyvale, CA, USA

P-78 **Increasing Extraction Efficiency of Pesticides & Dioxins from Wet Samples using a Novel New Polymer during Accelerated Solvent Extraction**
Sergio Guazzotti and Aaron Kettle, Thermo Fisher Scientific, Sunnyvale, CA, USA

P-79 **China as a Global Partner in Generation of Pesticide Residues Dataset**
Carmen Tiu, et al.; [1]Dow AgroSciences LLC, Indianapolis, IN , USA

P-80 **Bridging extraction efficiency information for data generation residue methods and QuEChERS enforcement methods using samples with in-grown residues—a case study**
Teng-yi Huang, et al.; Dow AgroSciences LLC, Indianapolis, IN, USA

P-81 **2α-Hydroxyursolic acid inhibits cell migration and invasion in MDA-MB-231 human breast cancer cells via EGFR-dependent PI3K/Akt signaling pathway**
Xue Jiang and Rui Hai Liu, Cornell University, Ithaca, NY, USA

P-82 **Washington State Department of Agriculture Chemical and Hop Laboratory workflow to ensure high quality data in single and multi-residue methods**
Kjersten Braaten-Fierros and Brittany Holmes, WSDA Chemical and Hop Lab, Yakima WA, USA

P-83 **Composting: A Biological Process for Aflatoxin Decontamination in Agricultural Environment**
Esther Y. Akoto, et al.; Department of Food Science and Technology, The University of Georgia, Griffin Georgia, USA

P-84 **Efficacy of soil amendments in reducing leaching of Agriculture Chemicals to soil and ground water in sub- tropical region of Uttarakhand, India - An Ecofriendly Approach**
Archana Suyal and Anjana Srivastava, Dept. of Chemistry - C.B.S.H, G. B. Pant Univ. of Agriculture and Technology, Pantnagar Uttarakhand, India

P-85 **Development of a FTIR Method to Identify Herbicides and their Low Volatile Counterparts**
Cedric Reid, et al.; [1]Mississippi State University, Department of Biochemistry, Molecular Biology, Entomology, and Plant Pathology, Mississippi State, MS

P-86 **Applying lipidomics for elucidating biomarkers and the role of environmental stressors leading to pansteatitis outbreak in fish across South Africa**
Jeremy P. Koelmel, et al.; University of Florida, Department of Chemistry, Gainesville, FL 32611, USA

P-87 **The determination of plasticizers and other chemical pollutants, from industrial point sources in Wallingford and North Haven with an emphasis on the pollutant's impact to indigenous fish populations in the Quinnipiac River.**
Harry Pylypiw, et al.; Department of Chemistry and Physical Sciences, Quinnipiac University, Hamden, CT, USA

P-88 **Use of a Multianalyte Method by Undergraduate Biology and Chemistry Students for the Analysis of Phthalate Esters and Industrial Contaminants in River Water**
Harry Pylypiw, et al.; Department of Chemistry and Physical Sciences, Quinnipiac University, Hamden, CT, USA

P-89 **Identification of Surfactants in Hydraulic Fracturing Fluids by Ion Mobility Mass Spectrometry**
E. Michael Thurman, et al.; University of Colorado, Environmental Engineering, SEEC, Boulder, CO, USA

P-90 **Analysis of Organic Marker Compounds and Hazardous Organic Compounds by GC/MS to Identify Contamination, Counterfeiting and Adulteration of Spices**
Patricia L. Atkins, SPEX CertiPrep, Metuchen, NJ, USA

P-91 **An Automated Extraction Solution for the Determination of Melamine and Cyanuric acid in Milk Based Products**
Tyler Trent, et al.; Teledyne Tekmar, Mason, OH, USA

P-92 **Time and Cost Effective Methods for Reducing Background Noise and Signal Suppression in Problem Matrices for Residue Analysis by LC-MS/MS**
Lisa Wanders, et al.; Thomson Instrument Company, Oceanside, CA, USA

P-93 **Quantitation of Terpenes in Cannabis Products Using LC-MS/MS and Atmospheric Pressure Chemical Ionization**
Paul Winkler, et al.; SCIEX, Redwood City, CA, USA

P-94 **Advantages of Non-Targeted Information Dependent Data Acquisition using LC-HR-MS/MS followed by Targeted Data Processing to Screen for Pesticides in Cannabis Samples**
Paul Winkler, et al.; SCIEX, Redwood City, CA, USA

P-95 **Using LC-HR-MS/MS and Intuitive and Automated Software Workflows to Quickly Identify Unknown Compounds in Food Samples**
Feng Zhong, et al.; SCIEX, Concord, ON, Canada

P-96 **Simultaneous Identification and Quantitation of Pesticide Residues in Food Samples using LC-HR-MS/MS**
Feng Zhong, et al.; SCIEX, Concord, ON, Canada

P-97 **Identification, Quantitation and Confirmation of Pesticides in Food Samples using LC-MS/MS and Ultra-fast Polarity Switching**
Farzad Pakdel, et al.; SCIEX, Redwood City, CA, USA

P-98 **A Highly Selective and Sensitive LC-MS/MS Method for the Quantification of Gluten Proteins**
Farzad Pakdel, et al.; SCIEX, Redwood City, CA, USA

P-99 **Quantitation of Glyphosate and Other Polar Pesticides in Beer Samples using LC-MS/MS**
André Schreiber, et al.; SCIEX, Concord, ON, Canada

P-100 **Identification of Artificial Colors and Dyes in Food Samples using LC-HR-MS/MS**
André Schreiber, et al.; SCIEX, Concord, ON, Canada

P-101 **Profiling of Hop-Derived Bitter Compounds in Beer using LC-HR-MS/MS and Statistical Data Processing**
Christopher Borton, et al.; SCIEX, Redwood City, CA, USA

P-102 **Qualitative Analysis of 12 Food Allergens in a Single LC-MS/MS Injection**
Christopher Borton, et al.; SCIEX, Redwood City, CA, USA

P-103 **Determination of Vitamin A and Vitamin E in Infant Formula**
Allen Misa, et al.; Phenomenex, Inc., Torrance, CA, USA

P-104 **Acrylamide from Coffee using Simplified Liquid Extraction (SLE) by HPLC**
Allen Misa, et al.; Phenomenex, Inc., Torrance, CA, USA

P-105 **Rapid and Simple Extraction and Analysis of Vitamin D2 and D3 from Dietary Supplements Using QuEChERS and HPLC-UV**
Scott Krepich, et al.; Phenomenex, Inc., Torrance, CA, USA

P-106 **Separation Solutions for Triglycerides in Food Fat and Oil by High Temperature GC Analysis**
Kristen Parnell, et al.; Phenomenex, Inc., Torrance, CA, USA

P-107 **Determination of Melamine from Pet Food using Liquid-Liquid Extraction and GC/MS Analysis**
Kristen Parnell, et al.; Phenomenex, Inc., Torrance, CA, USA

P-108 **Extraction of Cannabinoids from Brownies using QuEChERS Extraction and GC/MS Analysis**
Kristen Parnell, et al.; Phenomenex, Inc., Torrance, CA, USA

P-109 **A Rapid Screening Method for Analysis of Multi-Class Antibiotics from sausage using QuEChERS and LC/MS/MS**
Scott Krepich, et al.; Phenomenex, Inc., Torrance, CA, USA

P-110 **Analysis of Multiple Mycotoxins in Soybeans and Soymeal by LC-TOF: A Comparison of SPE and Modified QuEChERS Sample Preparation Methods**
Jason P. Weisenseel, et al.; PerkinElmer - Environmental Health, Shelton, CT, USA

P-111 **Gas chromatography coupled to triple-quad ICP-MS-MS for compound-independent quantification of organophosphate pesticides in honey bee products after miniaturized QuEChERS extraction**
Julio Landero, et al.; University of Cincinnati, Cincinnati OH, USA

P-113 **Do we still need to prepare matrix-matched calibration standards for GC-MS analysis of pesticide residues?**
Jack Cochran, et al.; Restek Corporation, Bellefonte, PA, USA

P-115 **Reduction in Pipette Tip Consumable Cost and Waste through Innovation**
Ali Safavi, Grenova LLC, Richmond VA, USA

ORAL PRESENTATION ABSTRACTS

A-1 The Evolution and Importance of Detection Methods In Pesticide Residue Analysis through the years

André de Kok

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The challenge for pesticide residue laboratories has always been to analyze as many pesticides as possible in multi-residue methods (MRMs), as fast, easy and cheap as possible, and at continuously decreasing limits of detection/quantification. Through the last 5 decades, this has been solved in a different, evolving way, depending on applied extraction and cleanup methods, but in particular depending on available detection systems. Nowadays, generic extraction methods, with minimum or no cleanup, can be combined with golden-standard, sensitive and selective GC-MS/MS and LC-MS/MS triple quadrupole detection methods. Scopes are possible up to 300-400 pesticides in one chromatographic run, depending on the instrument scan rate and the total chromatographic run time. Sensitivity, selectivity, scan rate and robustness of detectors are crucial parameters for the success of the total method. In this presentation, an overview of the gradual improvement of methodology over time, based on continuously improving detection systems, will be given. The latest achievements with recently acquired LC- and GC-HRMS detection systems will illustrate the present state-of-the-art.

O-1 The Analysis of Polar and Ionic Pesticides by Ion-Exchange Chromatography Tandem Mass Spectrometry; A Tale of Two (and many more) Molecules

Stuart Adams, Jonathan Guest and Mike Dickinson

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Ionic pesticides present many challenges to the modern analytical laboratory and are found in many areas of the food chain from glyphosate in beer to chlorate in milk. They are often not amendable to inclusion in the large multi-residue methods such as QuEChERS and have analytically challenging residue limits in international regulations. With the development of QuPPe (Quick Polar Pesticide) method the potential to analysis for these compounds has significantly increased. The QuPPe method covers a large range of polar and ionic pesticides but leads to matrix heavy extracts which can cause problems with retention time drift using reversed phased or HILIC chromatography, and therefore to cover many of the analytes several chromatographic runs are required.
At Fera we have coupled post column suppressed ion chromatography coupled with tandem quadrupole mass spectrometry (IC-MS) for the determination of ionic pesticides and metabolites in the QuPPe extracts. This approach has the potential to reduce the number of liquid chromatography runs required to cover a greater number of analytes. This presentation will describe our experience in IC-MS, the challenges and successes in the development, validation and implementation of this approach. Examples for various pesticide-matrix combinations will be discussed.

O-2 Evaluation of simultaneous MS and MS2 workflows of GC-LC-HRAMS for analysis of pesticides in fruits and vegetables

Łukasz Rajski,[1] María del Mar Gómez Ramos,[1] Sonia Herrera López,[1] Samanta Uclés,[1] Amadeo R. Fernández-Alba[1]

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During last decade high resolution accurate mass spectrometers –HRAMS- have improved qualitative (resolution, mass accuracy) as well as quantitative (sensitivity, linear range) aspects. HRAMS instruments are well known for their high selectivity in full scan MS mode. It is generally accepted that the simultaneous combination of full scan and MS/MS modes are necessary for the proper identification following the international Guidelines.
The objective of this work was to compare the performance of various workflows of simultaneous MS and MS2 analysis of fruit and vegetable samples. Evaluation covered all ion fragmentation (no precursor ion selection, ions from entire mass range are fragmented at the same time) and selected ion fragmentation (selection of precursor ion) named ddMS2, IDA, Auto MS/MS (depending of the platforms).
Detection and quantitation were carried out in full scan MS. MS2 data were used for identification.

Identification rates were close to 100% in practically each mode evaluated at 0.01 mg/kg. The higher selectivity of selection ion fragmentation mode offered in general better results for quantitation, being in the majority of the cases comparable to those obtained by GC-LC-QqQ-MS/MS. However a great advantage of all ion fragmentation was the possibility to enlarge the screening consequence of the non-targeted acquisition of the workflow. Practical cases and comparative results shows the high capability of these platforms for pesticide residue analysis in routine food control.

O-3 Targeted Discovery of Disinfection Byproducts in Swimming Pools and Spas

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Swimming pools are treated with disinfectants to protect swimmers from pathogens and prevent illness. The water used to fill a swimming pool if from a municipal drinking water supply, is also often treated with disinfectants such as chlorine. Disinfectants will react with naturally occurring organic matter in water and, in the case of swimming pools, they can also react with chemicals introduced to the water by the swimmers themselves to produce byproducts that can be potentially harmful. It is important to treat water while minimizing the risk of disinfection byproducts (DBPs). One of the first steps is to chemically characterize the DBPs in swimming pools and spas, very complex matrices, using discovery techniques since a lot of the contaminants are unknowns. Comprehensive two-dimensional gas chromatography high resolution time-of-flight mass spectrometry (GC×GC-HRTOFMS), was used for the tentative identification of “known unknowns” and “unknown unknowns” in swimming pool and spa water. The “known unknowns” were identified by library database searching deconvoluted spectra using LECO’s ChromaTOF software. The “unknown unknowns” were tentatively identified using a combination of EI and CI accurate mass data for chemical formulae determination and structural elucidation by leveraging the accurate mass fragments.

O-4 Strategies for Analysis of Pyrethroid Insecticides in Complex Matrices

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Synthetic pyrethroid insecticides have the advantage of low mammalian toxicity and are widely used for crop protection as well as in lawn and home consumer products, and also in lice control products for humans and companion animals. Extremely high hydrophobicity values and the corresponding tendency for these compounds to adsorb onto container surfaces present some more than typical difficulties for the pesticide residue chemist. Also, the instrumental screening method of choice for multianalyte pyrethroid analyses has traditionally been negative chemical ionization gas chromatography with mass spectral detection (NCI-GC-MSD), and there have been some additional challenges to overcome due to some inherent limitations (matrix effects and detector variability/drift) with this particular approach. One notable development over the past several years of environmental sample testing has been the emergence of d6 stable isotope analogues as a key tool in the generation of accurate and reproducible data. Techniques for analysis of biosolids samples from publicly-owned treatment works (POTWs) for eight synthetic pyrethroids will be presented as a case study. Challenges particular to this matrix include significant sample-to-sample compositional variability, along with the difficulty of finding control samples free of residues of all eight analytes. Both of these factors confound the residue chemist’s dilemma of what to use as a relevant control matrix for fortification recovery (quality control) purposes. The use of the d6 analogues both as instrumental internal standards (to normalize the NCI-GC-MSD responses) and as surrogates (to address the challenges of matrix background and sample-to-sample variability) will be presented and discussed.

O-5 A Multi-Residue Method for Monitoring Pesticides and Pesticide Degradation Products in Groundwater at Low Levels Using LC-MS/MS with On-line SPE

Agustin E. Pierri, Ph.D.

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With the ever-growing population and the increased agricultural activity in the last several decades, the domestic use of pesticides has increased drastically. As such, it becomes increasingly more important to monitor the fate and transport of these pesticides, especially in cases where agricultural activity can impact residential water sources—a vast majority of people who live in these areas rely on groundwater for drinking water. Since water can take up to

several decades to percolate through the earth to a deep aquifer, it is important to monitor both the pesticide as well as its degradation products. Owing to the diverse chemistry of these compounds, monitoring will undoubtedly require liquid chromatography coupled with tandem mass spectrometry. However, current analytical methods are based upon techniques that require time-consuming and tedious pretreatment such as extraction, concentration, and derivatization steps.
This presentation will describe a method for the determination of over fifty pesticides and degradation products down to 10 ng/L in untreated groundwater without sample pretreatment. By leveraging the extreme sensitivity of modern mass spectrometers and new developments in solid-phase extraction media designed for online-SPE, this method provides a robust solution for monitoring of a diverse group of compounds including herbicides, neonicotinoids, organo-nitrogen pesticides, and their degradation products at trace levels.

O-6 Development and Validation of a Large Multiresidue LC-MS/MS Method using On-Line Dilution and other Useful Features

Katerina Mastovska,[1] John Zulkoski,[1] Erika Deal,[2] Lukas Vaclavik,[3] Urairat Koesukwiwat,[4] Jean-Francois Halbardier,[3] Jerry Zweigenbaum,[5] and Thomas Glauner[6]

[1]**Covance Laboratories**, 3301 Kinsman Blvd; Madison, WI 53704, USA; katerina.mastovska@covance.com; [2]Covance Laboratories; Greenfield, IN, USA; [3]Covance Laboratories; Harrogate, UK; [4]Covance Laboratories; Singapore; [5]Agilent Technologies; Wilmington, DE, USA; [6]Agilent Technologies; Waldbronn, Germany

The aim of this work was to develop and validate a pesticide multiresidue LC-MS/MS method for the analysis of about 450 analytes eluting in less than 10 min. In addition to that, we wanted to improve retention and peak shape of early eluting, more polar analytes, which are notorious for having poor peak shapes when injected in extracts with a higher content of organic solvents, such as in QuEChERS acetonitrile extracts. We achieved that by using a special on-line dilution set-up (a serial combination of two high-pressure mixers), enabling an effective mixing of the injected sample with the initial highly aqueous mobile phase before reaching the column. We also included in-run blank injections as an effective tool for reduction/prevention of carry-over. The method employs triggered multiple reaction monitoring (tMRM), which allows triggering of additional MRMs when one of the primary MRMs exceeds a set threshold. We included 2-3 primary MRMs and typically four or more total MRMs, resulting in > 2000 MRMs in the method. The mobile phase gradient was optimized using MRM histograms to spread the analytes and MRMs evenly throughout the elution window, with special attention being paid to the separation of critical pairs. The method was validated in three different laboratories in multiple commodity types/matrices using SANTE method validation guidelines and criteria, with 0.01 mg/kg method validated LOQ achieved for the majority analyte-matrix combinations. The presentation will discuss development, performance and inter-laboratory transfer of the method, sharing various practical optimization and routine use tips and tricks.

O-7 Multi-lab Validation Study of 204 Pesticides in Fruits and Vegetables by QuEChERS and LC-MS/MS

Kelli Simon[1], James Wittenberg[1], Jon W. Wong[1], Kai Zhang[1], and Paul Yang[2]

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A multi-laboratory (n=3) validation study employing a liquid chromatography-tandem mass spectrometry (LC-MS/MS) multiresidue method for the measurement of 204 pesticides is presented. Vegetation samples are extracted by the *Qu*ick, *E*asy, *Ch*eap, *E*ffective, *R*ugged and *S*afe (QuEChERS) procedure, diluted and filtered prior to analysis via LC-MS/MS. All laboratories employed previously comminuted and homogenized broccoli, orange and carrot in addition to local produce that are of interest to a laboratory. All laboratories employed an ABSciex iDQuant Standards Kit for Pesticide Analysis, Restek Q-Sep QuEChERS Kits and an ABSciex Qtrap 5500 mass spectrometer. LC separation was performed using a Restek Ultra-Aqueous C_{18} Column, 10 mM NH_4HCO_2 buffer (A: water, B: methanol) with 0.1% formic acid and 0.5 mL/min flow rate. The ruggedness of the method is established through individual laboratory impacts on the standard operating procedure for sample preparation, LC and MS/MS parameters. The percentage of analytes with LOD < 1 ng/g is 89-97% and 78-97% of analytes have $R^2 > 0.99$. The percentage of analytes with MDL < 1 ng/g ranged from 89-99% with less than 5% intralaboratory variation across all matrices evaluated. At least 85% of analytes met the acceptance criteria (70% < % Recovery < 120%; <20% RSD) for broccoli, orange and carrot at three fortification levels. Similar performance was obtained for grape, pomegranate and blueberry where 10% less analytes met the acceptance criteria for wheat flour and raisin. This method was applied to incurred broccoli and orange where up to seven pesticides were detected above 1 ng/g.

O-8 Multi-Class, Multi-Residue Veterinary Drug Analysis in Infant Formula and Related Ingredients Using UHPLC-MS/MS

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A multi-class, multi-residue method based on UHPLC-MS/MS was developed for the analysis of around 150 compounds belonging to a variety of veterinary drug classes: anthelmintics, antibiotics (amphenicols, beta-lactams – penicillins and cephalosporins, lincosamides, macrolides, quinolones, sulfonamides, tetracyclines, and others), antimicrobial growth promoters, antiprotozoals, beta-agonists, coccidiostats, dyes, selected insecticides, tranquilizers and others. The method development and optimization was divided into five main phases: (i) MS/MS conditions for individual compounds; (ii) LC conditions; (iii) final UHPLC-MS/MS method; (iv) sample preparation procedure; and (v) method validation, data acceptance criteria, and method implementation. The particular attention was devoted to the comparison of different sample preparation approaches and to optimization of the mobile phase composition to achieve a well-distributed analyte elution profile, minimum interferences, and optimum sensitivity. The sample preparation optimization was divided into three stages: (i) extraction procedure; (ii) different clean-up options (such as hexane defatting, dispersive SPE clean-up, or supported liquid extraction); and (iii) establishment of the sample extract dilution scheme. The method consists of dispersing the sample in water with an EDTA buffer, followed by extraction with 0.1% formic acid in acetonitrile. The analytical determination is performed by UHPLC-MS/MS using positive electrospray ionization mode. The method was validated in infant formula powder and its ingredients at six spiking levels (0.5, 1, 5, 10, 50, 100 ng/g). Analyte LOQs were set at the lowest spiking levels that consistently met acceptance criteria (trueness and repeatability) established based on the CAC/GL 71-2009 guideline document.

O-9 Targeted Multiresidue Analysis of Veterinary Drugs in Dairy Products Using Liquid Chromatography-Tandem Mass Spectrometry (LC-MS/MS)

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The FDA is required to screen and regulate dairy products for veterinary drug residues. Although there are currently analytical methods available for analyzing veterinary drugs in dairy products, namely milk, this project aims to improve on these methods by utilizing technological advancements as well as increasing the number of additional veterinary drugs monitored. This research focuses on the development of validated methods for detecting and quantifying up to 60 diverse veterinary drug residues in a variety of dairy products using current technologies in order to complement and/or replace older methods. Currently, two chromatographic runs are required to analyze all 60 compounds due to polarity differences. The first method for the nonpolar compounds involves an acetonitrile/water extraction with additional solid-phase clean-up for high-fat dairy matrices followed and subsequent analysis by LC-MS/MS. Preliminary results indicate that 40 out of 52 nonpolar target compounds reach acceptable recoveries (70–120%, RSD<20%) at three fortified levels in milk powder. A second method, based on Drake *et al.* (LIB #4589), is used for the analysis of 8 polar compounds, primarily aminoglycosides. This involves a water extraction followed by solid-phase clean-up and subsequent analysis by HILIC-MS/MS. With these new methods in place, the FDA will be able to better monitor various dairy food products for a wide range of veterinary drugs.

O-10 Development and validation of a screening method for drug residues in fish and shrimp using liquid chromatography high resolution mass spectrometry

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A screening method for veterinary drug residues in fish and shrimp using LC with a Q-Exactive MS instrument has been developed and validated. The extraction procedure and MS acquisition parameters were optimized with 60 validation compounds representing a variety of veterinary drug classes. The sample preparation procedure consisted of an acidic acetonitrile extraction followed by solid phase extraction cleanup. Initially data were collected using nontargeted acquisition (MS^1 and MS^2 with All Ion Fragmentation). Residues were detected based on the exact mass of precursor and

product ions, along with isotope pattern and retention time matching. Semi-quantitative data analysis compared peak areas from MS^1 signal to a one-point extracted matrix standard at a target testing level. All validation compounds could be detected and confirmed in salmon, tilapia, catfish, and shrimp extracts fortified at the target testing levels; many analytes could be detected at much lower levels. Fish dosed with selected analytes and aquaculture samples previously found to contain residues were also analyzed. The screening method could be expanded to monitor for ~250 additional veterinary drugs based on exact mass measurements and retention times. Data were also obtained using a more targeted acquisition program (data dependent MS^2) to obtain additional qualitative information.

O-11 Important Considerations with Regard to Sample Preparation when Developing Reliable Analytical Residue Methods.

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With the availability of sensitive and selective instrumentation such as mass spectrometry, proper use of sample preparation is often times overlooked. For example, since the pesticides registered over the last decade are mostly polar compounds that are not amenable to gas chromatographic techniques, LC-MS/MS is the methodology of choice to monitor these contaminants in foods for enforcement purposes. Likewise LC-MS/MS is needed for the reliable determination of other polar food contaminants such as mycotoxins, veterinary drugs, and melamine and its analogs. Although LC-MS/MS offers a number of advantages in terms of sensitivity, selectivity and overall speed of analysis, there are a number of important considerations which must be kept in mind when developing analytical methods that rely on this technology. Some of these critical components include: extractability; test portion cleanup; specific chemical properties of analyte such as polarity and pH of analyte; choice of internal standards and how to properly use them; spiking of test portion versus incurred residue analysis; ruggedness; and the use of standard reference materials (if available) and/or proficiency test samples. The impact of these critical components on method development will be discussed using well known examples of challenging problems such as the analysis of over 200 polar pesticides in foods and dietary supplements, mycotoxin analysis, plus other examples of current interest.

O-12 Identification of Penicillin G Metabolites under Various Environmental Conditions using UHPLC-MS/MS

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In this work, we investigate the stability of penicillin G in various conditions including acidic, alkaline, natural acidic matrix, and after treatment of citrus trees that are infected with the citrus greening disease. The identification, confirmation, and quantitation of penicillin G and its various metabolites were evaluated using two UHPLC-MS/MS systems with variable capabilities (i.e. Thermo Q Exactive Orbitrap and Sciex 6500 Qtrap). Our data show that under acidic and alkaline conditions, penicillin G at 100 ng/mL degrades fast, with a determined half-life time of approximately 2 hours. Penillic acid, penicilloic acid, and penilloic acid are found to be the most abundant metabolites of penicillin G. These major metabolites, along with isopenillic acid, are found when penicillin G is used for treatment of the citrus greening infected trees. The findings of this study will provide insight regarding penicillin G residues for food safety purposes, in agricultural and biological applications.

O-13 Comparison of Sample Preparation Techniques and Screening for >120 Veterinary Drugs in Animal Meat

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Veterinary drug (VD) mismanagement or overdose when trying to control diseases in livestock can lead to residues being present in processed animal meat prepared for human consumption. A critical step before analysis is the extraction of these VDs with disparate chemical properties. In this study, two sample preparation and extraction techniques were evaluated in a cross-lab validation. The traditional and widely used QuEChERS with dispersive-SPE extraction procedure employed at the USDA-ARS was compared against a new lipid removal material, Enhanced Matrix Removal (EMR)-Lipid method. The study involved the comparison of both methods for extracting >120 VDs spiked at the maximum tolerance levels in bovine kidney, muscle and liver. Sample extracts were then evaluated using a veterinary drug library with

accurate mass, MS/MS spectra and retention time confirmation on a liquid chromatograph coupled to a quadruple time-of-flight mass spectrometer (LC-QTOF).
The results of this study indicated that both methods are well suited for extraction of a wide variety of VDs from bovine meat. While some specific compound classes had differences in recoveries between the methods and will be illustrated, overall recoveries were acceptable (70-120%) in all three bovine matrices. Other parameters evaluated in this validation study included co-extractive amount, absolute recoveries and matrix effects for the VDs tested. Further, the ability to do sensitive quantification at the ng/g level using the LC-QTOF was also evaluated. The results from this study indicate that there are simple, robust and viable methods available for the extraction of VDs in meat.

O-14 Pesticide residue analysis in Hops: analysts' nightmare or a unique opportunity to advance

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Hops is considered the green gold among the brewery raw materials and the cost of this key ingredient is related to the yield per plant. The range of problems that can arise in growing the hop plant includes bacterial diseases, fungus and mildew, virus diseases, as well as pests and parasitic invasion. In fact the hop plant belongs to crops with intensive chemical protection.
Today, three primary sample preparation methods abbreviated as "QuEChERS" have been established for a wide scale of matrices such as fruits and vegetables and, in combination with dispersive solid phase extraction (dSPE), are also applicable for matrices like fatty matrices or black tea. But these methods are hardly applicable to hops without further modification due to co-extraction of matrix components (chlorophyll, resins, bitter acids) together with compounds of interests, which can most often cause massive signal suppression, elevated background, and other negative matrix effects. Unlike the matrices such as fruits or vegetables, only two suitable sample preparation protocols were published for hops matrix. These methods utilize acetonitrile or acetone extraction, solid phase extraction and liquid chromatography with tandem mass spectrometry (LC-MS/MS).
In our initial set of experiments we focused on developing a QuEChERS based method in combination with a new generation of dSPE sorbent blends. Our attention then focused on comparing the matrix effects of this method with previously described sample preparation procedures. In addition, we attempted to compensate matrix effects using extensive extract dilution as a valuable tool to overcome the problem of signal suppression.

O-15 Addressing Consumer Complaints Samples in a Regulatory Laboratory; Food Borne Illness Investigations and Consumer Fraud

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Between 2005 and 2013 the Florida Food Safety Laboratories receive more than 400 samples that were recorded as "consumer complaints". The complaint can be anything from the product looked, tasted or smelled odd to the food product was thought to cause physical discomforts that may indicate mild forms of food poisoning. These symptoms might include, nausea, diarrhea, headache, a tingling or burning of the mouth. Other complaint samples indicate possible food fraud where a product is mislabeled which can be a health concern and an economic concern.
The laboratory sample is picked up by an inspector from a retail outlet with the same lot number as the sample that the consumer had issue with. Most complaint samples undergo organoleptic testing. Our microbiology performs the same analysis that they would routinely perform on the commodity in question. The chemistry section typically analyzes the samples for metal contamination and may assign a test that seems appropriate for the commodity and complaint unless our customer, either Florida Department of Health (FDOH) or our inspection group, requests a specific test. In that case only the requested test is performed.
Harmful contaminants are usually not identified. These negative results could be due to a number of reasons including that the food tested was not what made the consumer ill or the packaging on the consumers sample had been compromised. In this presentation we will examine various strategies that may improve the likelihood of finding a contaminant in these samples.

O-16 Establishing the Minimum Analytical Method Validation Procedure for EPA FIFRA Misuse Samples to Meet the ISO/IEC 17025:2005 Requirements

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Under the Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA), the U.S. Environmental Protection Agency (EPA) grants the authority over regulating pesticide use to each state. Samples for pesticide misuse cases such as drift complaints are analyzed by state laboratories (state FIFRA labs). The major challenges the state FIFRA labs face are: 1) Sample matrix and analyte combination is unpredictable, 2) A quick turnaround is required. Consequently, the majority of the samples are analyzed by non-standard methods that are developed by the laboratory based on the nature of the sample. It is practically impossible to pre-validate such non-standard methods. Yet, the FIFRA enforcement samples have forensic nature and the results of the analyses are often brought to court. The analytical results have to be legally defensible.
The author's laboratory (the MDA lab) developed the minimum method validation procedures for the non-standard methods and sought ISO/IEC 17025:2005 accreditation for the methods used to analyze the FIFRA enforcement samples. The accreditation was granted to the MDA lab in May 2015 under the Flexible Scope Approach by A2LA. The developed validation protocol as well as the procedural changes made to the daily operations at the MDA lab are presented. The pros and cons of the ISO/IEC 17025:2005 accreditation for the state FIFRA labs are also discussed.

O-17 The Pesticide Data Program – 25 Years (and counting)

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The Pesticide Data Program (PDP) administered by the U.S. Department of Agriculture (USDA), Agricultural Marketing Service (AMS) enters its 25th year. The pesticide data that USDA publishes each year provide regulators, scientists, farmers, processors, manufacturers, and consumers with important insights into the actual levels of pesticide residues found on widely consumed foods. It has been said that PDP is the gold standard of monitoring programs. This presentation provides a look back at the history and a look forward into the future and shows that PDP continues to evolve as a program while still providing high-quality data for dietary risk assessments and demonstrating that the pesticide levels in the U.S food supply are below the allowable levels. PDP is a Federal-State partnership and tests a wide variety of domestic and imported foods using a sound statistical program and the most current laboratory methods.

O-18 Ongoing Method Development and Harmonization Efforts at FDA Pesticide Monitoring Laboratories

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Several changes to the FDA pesticide monitoring program are in the works. Effort is being made to collaborate and harmonize methods across FDA labs and expand the scope of analysis to new/more residues, including a few problematic ones like acid herbicides and glyphosate. High resolution mass spectrometry (HRMS) is being evaluated as a replacement for existing LC-MS/MS determination procedures. A QuEChERS-based extraction and new cleanup protocol for GC analysis has also been implemented at most FDA labs and collaboration efforts for it are underway. In addition, a negative mode-only LC-MS/MS procedure is being developed. Each of these projects will be discussed briefly. How these projects mesh with existing program resources, methods, instrumentation and goals will be considered.

O-19 Freshwater and Marine Algal Toxins: An Old Problem with Expanding Consequences

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Algal toxins in freshwater, brackish, and marine systems are well-known environmental contaminants and for the most part are well characterized in terms of the number of known toxins and syndromes. With a few exceptions (such as BMAA) new toxins and syndromes are rare, and we have made great strides in understanding the evolutionary

and molecular basis for production of many of these compounds. Despite this knowledge base, there is increasing awareness that "unusual" toxin events are becoming more common, driven in part by climate change, globalization, and increased awareness that many of the algae (and therefore toxins) are more ubiquitous than previously thought. For example, in the past few years the U.S. has experienced the largest ever recorded domoic acid event in the marine waters of the eastern Pacific, massive impairment of drinking water due to freshwater algal blooms in Lake Erie, and expansion of (aerosolized) brevetoxins from marine dinoflagellates throughout the Gulf of Mexico. These events are symptomatic of global expansion of algal toxins. Obvious questions that arise from these events are whether (and why) we will see continued expansion of blooms to new regions, what the consequences are of exposure to multiple toxins simultaneously, and what we should do about it. This resentation will document some of these events and the potential consequences, highlighting new technologies and national/international programs designed to understand, predict, and potentially mitigate the consequences of this global expansion.

O-20 Evaluation of Methods for Extraction of Tetrodotoxin and Saxitoxin from Fresh and Salt-Dried Puffer Fish with LC-MS/MS Analysis

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The marine toxin Tetrodotoxin (TTX) has been implicated recently in several non-fatal poisoning incidents in the United States. It has become a priority for the FDA to have a validated method for detection and quantification of TTX in place, to ensure that the food supply remains safe and to corroborate instances of illegal importation of puffer fish. We have developed an LC-MS/MS assay capable of quantifying TTX from 5 -250 ppb utilizing UPLC and HILIC chromatography coupled with MRM mass spectrometry. Our goal is to optimize the detection of TTX in fish extracts at concentrations at least 10x below the anticipated safety level, as there is no regulatory limit on the amount of TTX in seafood. This method is also capable of distinguishing TTX from Saxitoxin (STX), a neurotoxin of similar size and potency, which can co-occur with TTX in marine and freshwater fish and is regulated at 800 ppb. Extraction of TTX and STX from fish matrices has presented a difficult challenge and efforts are underway to develop efficient sample preparation protocols for both fresh and salted/dehydrated fish fillets. The high salt content in dehydrated fillets must be mitigated as it can interfere with HILIC analysis. Additionally, the large quantity of collagen and other proteins co-extracted with the toxins in fresh puffer fillets can impede further downstream sample processing. Discussion of sample preparation approaches for concentration and purification of TTX, STX, and their related metabolites extracted from fish will be presented.

O-21 Detection of Paralytic Shellfish Toxins and Domoic Acid by LC-MS/MS

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Consumption of shellfish contaminated with marine biotoxins can cause severe intoxications in humans, which can be different depending on the toxins involved in the contamination. Biotoxins have been classified into eight groups, including domoic acid that causes Amnesic Shellfish Poisoning (ASP) and saxitoxin and its derivatives that cause Paralytic Shellfish Poisoning (PSP). In the EU, these biotoxins are regulated by legislation which limits the concentrations of biotoxins in seafood placed on the market. Production areas are subjected to periodic controls intended to check for biotoxins. The EU specifies analytical methods to be used for official control. Those for ASP and PSP toxins both use HPLC. The latter is a complex and lengthy method based on pre-column oxidation and fluorescence detection.
In recent years, focus has shifted to highly sensitive and specific LC-MS/MS methods. Turner *et al.* (2015) reported the single-laboratory validation of a method for PSP toxins, originally developed by Boundy *et al*. (2015), using solvent extraction, SPE and HILIC LC-MS/MS. In contrast, the aim of this project was to develop a quick and robust screening method, also based upon LC-MS/MS but without SPE, for analysis of shellfish samples for ASP and PSP toxins: domoic acid, saxitoxin, neosaxitoxin, decarbamoylsaxitoxin, decarbamoylneosaxitoxin, gonyautoxins 1-6, N-sulfocarbamoyl gonyautoxins 1-4 and decarbamoylgonyautoxin 2-3. The method was successfully validated (Decision 2002/657/EC) and tested with materials from EURL-MB Proficiency Tests. When implemented during routine surveillance, any samples shown to contain biotoxins that exceed the screening target concentration would be submitted for confirmatory analysis using the official methods.

O-22 Pesticide Residues in Human Milk: ppb detection in 1 mL

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A method has been established which screens for over 80 pesticides in human milk with a detection limit of 1 ppb using 1mL of human milk.
As part of the development process the matrix enhancement has been determined for each residue in a QuEChERS extract and analyte protectants have been used to standardize the enhancement in milk extract and standards in solvent. Development of a pressure pulsing method has improved the limit of detection substantially. A literate QuEChERS based sample preparation method has been scaled from 10 mL to 1 mL of human milk.
The net result is that the pesticide residues in human milk can be detected at levels of 1ppb or better and the use of analyte protetctants makes quantification very reproducible. The fat content of the milk has been used as a normalizing factor for reporting of levels.
The method has been used for geographical cross section studies on over 40 mothers and longitudinal studies on 16 mothers. The levels detected in this study have been very low and will be presented in the context of world levels together with future plans.

O-23 Contaminant Testing in Marijuana: Pesticides, Mycotoxins and Residual Solvents

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The use of marijuana and marijuana extracts for medicinal purposes has seen increased acceptance and approval in both North America and Europe. In addition to cannabinoids, the chemical composition of marijuana includes sugars, hydrocarbons, some proteins, fatty acids, phytosterols, terpenes, phenols, and flavonoids. This matrix complexity presents a challenge to laboratories faced with developing methods for the analysis of contaminants such as mycotoxins and pesticides. This presentation will present simple and effective sample preparation and analysis methodologies for three areas of interest in marijuana contaminant testing: pesticide residues, aflatoxins and residual solvents. The marijuana used for method development was procured from the Chemistry & Physiological Systems Research Branch of the National Institute on Drug Abuse at the National Institute of Health. Pesticides were analyzed by GC/MS/MS after QuEChERS extraction and cleanup using a new sorbent blend designed to remove pigment while increasing recovery of planar compounds. Aflatoxins were analyzed by LC/MS/MS after liquid extraction and cleanup utilizing a solid phase extraction cartridge designed for cleanup of mycotoxin extracts. Residual solvents were analyzed from hemp extract using a simple headpsace solid phase microextraction method followed by GC/MS. For all three applications, details of the methodologies will be presented along with data showing accuracies and reproducibilities for the analysis of spiked replicate samples.

O-24 Navigating the Pesticide Related Regulatory Landscape With Respect to Individual State Legal *Cannabis* Cultivation in the US.

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The current regulatory environment around pesticide use for individual state legal *Cannabis* production in the US is complicated by the multitude of State and Federal Laws that are in play. This presentation will hopefully shed light on safety assessment concerns and on the ways analytical chemistry can help mitigate these concerns. Currently, no pesticide labels for agrochemical use on *Cannabis* have been approved under the Federal Insecticide, Fungicide and Rodenticide Act (FIFRA), the federal laws governing pesticide usage. Thus legal growers have very few, if any, approved options for pesticide use during production. Pesticides generally used in commercial food and ornamental production agriculture are clearly prohibited as growers must adhere to strict label requirements. Therefore growers have resorted to over the counter or organic pesticide treatments. Many analytical laboratories routinely analyze for these pesticides

products in food and feed applications but are just beginning to evaluate the analytical needs of the *Cannabis* industry. Logistical, legal, and ethical concerns surrounding pesticide analytical support for this industry must be considered if the safety of these products from a pesticide exposure perspective is to be addressed.

O-25 Pesticide Analysis in Highly Resinous Matrices: Options for Sample Preparation and Cleanup Prior to LC-MS/MS and GC-MS/MS Analysis

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In recent years, food safety laboratories have adopted new and simplified analytical methods designed to reduce analysis time and related costs, as well as to increase throughput. Simplified procedures, such as QuEChERS combined with dispersive SPE (dSPE) cleanup, require only minutes for sample preparation compared with prior methods that took hours or days. Also, the availability of more sensitive and selective instrumentation allows for hundreds of compounds of many classes to be determined in a single injection compared with the traditional compound or class specific approach. After the initial QuEChERS extraction, a simple and fast cleanup procedure is desirable to reduce matrix contaminants potentially detrimental to instrument performance and analytical column lifetime. Cleanup options will be discussed for pesticide analysis in a number of natural products including spices and cannabis. These types of matrices contain a multitude of resinous constituents often very similar in physicochemical properties to many pesticides. Because these interfering resin constituents are so similar in solubility and polarity to the target analytes, typical dSPE cleanup protocols used for fruits and vegetables may not be suitable for pesticide analysis in dried natural product matrices. After cleanup, pesticide analysis is performed using tandem LC-MS and tandem GC-MS. A new GC-MS technique using atmospheric pressure mass-spectrometry (APGC) is employed for the tandem GC-MS analysis.

O-26 Poisoned Garden: Pesticides in Cannabis by modified QuEChERS and LC-MS-MS

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Legalization of medical and recreational marijuana (*cannabis spp*.) in some areas has resulted in tremendous growth in cultivation of this potent plant. Some growers have aggressively applied chemicals to increase yield of this high value and somewhat vulnerable crop. To protect the public, sensitive and selective methods for residue detection are needed. We developed a high performance method for determination of almost 200 chemical residues in dried cannabis flower and cannabis concentrates. Over 100 cannabis dried flower and concentrates offered for retail sale have been analyzed. Statistics of our market survey will be presented along with a description of the unique challenges we faced from the cannabis matrix. Ground and homogenized cannabis was extracted with a modified QuEChERS and dispersive SPE cleanup. Detection was carried out by UHPLC-MS-MS with continuous polarity switching. Pesticide recovery was determined using spiking experiments and matrix-matched calibration curves. Matrix matched calibration curves were linear within the quantitation limits established for each compound, ranging from as low as 1.5 ng/g to 2000 ng/g at the upper limit. Quantitation limits had less than 20% RSD in triplicate, sufficient signal to noise, and freedom from interference in multi-lot matrix tests. Recovery was compound dependent but nearly all were within the range of 70-120%. Analysis of dried cannabis flower and cannabis concentrates revealed widespread presence of residues at a wide range of concentrations. Cannabis concentrates often had the highest levels, extending from tens to hundreds of mcg/g levels reflecting possible concentration during the extraction process.

O-27 Direct Determination of Glyphosate, Glufosinate, and AMPA in Food by Liquid Chromatography/Tandem Mass Spectrometry

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Glyphosate is the most applied herbicide in the world due to its high efficacy, low toxicity and affordable price. It is too polar to be recovered by a QuEChERS extraction and too polar to be retained by a reversed-phase HPLC. Historically, a time-consuming precolumn derivatization of glyphosate with 9-Fluorenylmethyl chloroformate (FMOC-Cl) was used to enable the retention of glyphosate on the reversed-phase column. Direct determination of glyphosate methods on

many specialty columns such as anion, HILIC, and mix-phase mode columns were also used with limited success. A simple high-throughput liquid chromatography/tandem mass spectrometry (LC-MS/MS) method was developed for the determination of glyphosate, glufosinate and aminomethylphosphonic acid (AMPA) in different food matrices using a reversed-phase with weak anion-exchange and cation-exchange mixed-mode Acclaim™ Trinity™ Q1 column. Soybean, corn, egg, milk, and honey were shaken with an aqueous solution of acetic acid and ethylenediaminetetraacetic acid disodium salt for 10 min. After centrifugation, the supernatant was passed thru an Oasis HLB SPE to retain suspended particulates and non-polar interferences. The sample was directly injected and analyzed in 6 min by LC-MS/MS with no sample concentration or derivatization steps. The method will be used in the FY 2016 FDA pesticide screening program.

O-28 Swedish Multiresidue Method SweEt goes into Orbitrap Technology

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In a world of constantly new produced pesticides, the use of full scan technology has become very important. To be able to find and measure these compounds, not defined before analysis, is the major advantage and difference from analysing them with triple-quadrupol instruments.

Much has happened with the development from the first generation of high resolution instruments which were both expensive and complex to learn. They are still very advanced but the manufacturers have made a great effort in making the software easy-to handle with a reasonable price tag.
The procedure of today at Swedish National Food Agency has been to analyse the validated scope with around 300 analytes with triple quadrupole and then searching for unknowns in a different sequence with HRMS. The aim of this work was to introduce the Q Exactive Focus Orbitrap as a routine instrument for analysing pesticides in fruits and vegetables with around 300 analytes and simultaneously search for new unknowns. Combining UPLC with full scan MS and simultaneously measure unlimited amount of analytes gives enormously possibilities.
This presentation will discuss the possibilities of introducing the Q Exactive Focus Orbitrap as a routine instrument according to the European guidance document, SANTE/11945/2015, criteria for screening, identification and quantification as well as the easy-to-handle for laboratory technicians.
We will also describe the workflow for targeted and non-targeted analyses as well as the pros and cons.

O-29 Using Sandwich Injections to add Matrix, Internal Standards and/or Analyte Protectants for the GC/Q-TOF Analysis of Pesticide Residues

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It is well known that certain pesticides interact with “active sites” in the GC or GC/MS flow path causing losses due to adsorption or decomposition. This is most notable when calibration standards prepared in solvent are used to quantify real samples containing matrix. For this reason calibration standards are usually prepared in clean matrix or matrix with analyte protectants (APs). The matrix and APs out-compete pesticides for these active sites, resulting in higher pesticide responses. APs serve as an artificial matrix that can substitute for matrix or be added to the matrix to reduce analyte losses. But, adding analyte protectants to each vial is an extra sample preparation step and many labs do not use them. This presentation looks at ways to automate the addition of APs and/or matrix by making sandwich injections. The autosampler was able to draw from up to three different vials, so solvent standards were injected with various combinations of APs, matrix and matrix with added internal standards. As predicted, significant response enhancement was observed when a solution of APs or APs and matrix were injected together with analytes in solvent. Experiments were conducted to see if there was a difference between having the APs on the top or bottom of the sandwich. There was some advantage to having the APs on the bottom, presumably because they enter the inlet first and can interact with some of the active sites before the analytes arrive.

O-30 Profiling Maple Syrup for Authenticity and Adulteration by Chemical Composition using UHPLC/QTOF MS

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Maple syrup, a condensate of maple tree sap, contains at least 65% sucrose. It requires boiling 40 gallons of sap to produce one gallon of syrup. Although Vermont produces the most maple syrup in the United States, worldwide the greatest producer is Canada with farms in Ontario and Quebec. Northeast states also produce it including NY, ME, MA, CT, PA, and NH with small production Maryland. Both the United States and Canada grade syrup similarly based on the time of year the sap is collected. Grade A fancy from Vermont is similar to Claire in Quebec and both saps are collected in early spring. They are very light in color where grade B is darker and collected in later spring. Given grade and region, it would appear verification of maple syrup to be difficult. Analysis of the syrup by extraction using C18 SPE and elution with methanol followed by positive ion UHPLC/QTOF MS shows a complex profile of compounds. Direct analysis (filtering only) of real and imitation maple syrup easily distinguishes the two. Interestingly, the positive ion data for maple syrup is very similar regardless of region or grade. In contrast, the negative ion UHPLC/QTOF MS data does show distinction of by grade whereas region is more complex. The determination of authenticity, verification and adulteration requires data mining and multivariate statistical approaches. This work examines the chemical profiling of syrups from different regions and grades and identification of marker compounds for real maple syrup.

POSTER ABSTRACTS

P-1 Improved Cleanup for the LC/MS/MS and GC/MS/MS Analysis of Pesticides In Turmeric Powder Using a Novel Dual-Layer SPE Cartridge

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The complex composition of turmeric powder presents a challenge when performing chromatographic analysis of pesticides. Residual pigments and oils from the turmeric matrix can contaminate both GC/MS and LC/MS systems, and also result in significant matrix effects. Therefore, an effective cleanup method is needed prior to the injection of turmeric extracts onto GC or LC systems. Standard QuEChERS methodology does not offer the cleanup capacity required for the analysis of high background samples such as turmeric. Recently, a novel dual-layer cartridge was designed for the cleanup of acetonitrile extracts from difficult matrices such as dry commodities (spices, tea, etc.) prior to pesticide residue analysis. The top bed consists of a mixture of PSA, C18 and a novel graphitized, spherical carbon. This carbon was engineered to remove sufficient pigmentation while allowing for better recoveries of planar compounds, without the need for toluene in the elution solvent. The bottom layer of the cartridge contains a zirconia-coated silica. This sorbent removes oily residues and provides additional retention of some pigments. This study demonstrates the use of this unique dual-layer SPE cartridge for the cleanup of turmeric powder extracts prior to pesticide analysis by GC/MS/MS and LC/MS/MS. Turmeric powder was spiked at 100 ng/g with a wide range of pesticides of different polarities and classes. Quantitation was performed against multi-point calibration curves prepared in unspiked turmeric extract (after cleanup). The recovery, reproducibility, and background removal in the analysis of 51 different pesticides from turmeric extract will be discussed.

P-2 Planar Pesticide Quantitation from Green Food Matrices Using QuEChERS Cleanup With a Novel Graphitized Carbon Black and Zirconia-based Adsorbent

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Pesticides with planar structures, such as hexachlorobenzene and chlorothalonil, are commonly used during the cultivation of spinach and other leafy, green crops. The extraction and analysis of these residues is often complicated by the presence of chlorophyll in green food matrices. In both GC/MS and LC/MS/MS analyses, large pigment molecules, such as chlorophyll, are highly problematic. They can accumulate in the inlet and degrade column performance in GC/MS. Chlorophyll is also known to contaminate the LC/MS/MS source. Method EN15662 recommends the use of Graphitized Carbon Black (GCB) in sample cleanup methods for the removal of chlorophyll and other pigment interferences. While traditional GCB efficiently removes pigment molecules, it also retains planar pesticides as a result of π-π interactions between these compounds and the graphitic carbon. This presentation features a unique QuEChERS cleanup mix that contains a zirconia-containing adsorbent in combination with primary-secondary amine (PSA) and a proprietary GCB . The GCB used removes >95% the chlorophyll from green matrices while providing recoveries ranging from 70% to 120% of challenging planar pesticides. The use of the sorbents to remove chlorophyll while maintaining high recovery of planar and non-planar pesticide residues in spinach and other green matrices will be discussed. The effects of GCB quantity and surface area on color removal and pesticide recovery will be examined. Color removal from QuEChERS extracts of spinach was determined by visible spectrophotometry measurements, and pesticides recoveries (planar and on-planar) by GC/MS/MS.

P-3 Solventless Extraction and Analysis of Pesticides in Foods by Direct Immersion SPME Using an Overcoated Fiber

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Solid phase microextraction (SPME) is a solvent-less extraction technique that is easily amenable to automation. Sample extraction can be done either in headspace or by direct immersion. The SPME approach chosen is often determined by the sample matrix and analyte class. In the case of pesticide analysis, direct immersion is often used due to the low vapor pressures of these compounds. However, direct immersion of standard SPME fibers into complex matrices such as foods can prematurely foul the fiber, leading to loss in analyte response. In this work, we utilized a new polydimethylsiloxane (PDMS) overcoated version of a PDMS/divinylbenzene (PDMS/DVB) SPME fiber for analysis of

pesticides from a complex food matrix. The PDMS overcoating serves as a barrier between the adsorbent DVB layer and the matrix, but allows smaller analytes to migrate through and be retained. The overcoating increases the life of the fiber by minimizing adhesion of matrix to the SPME coating and by improving physical durability. Data will be presented in which this new overcoated fiber was used for extraction of a set of select pesticides from spaghetti sauce prior to GC/MS analysis. The SPME method was optimized to enhance sensitivity, accuracy and reproducibility. Performance of the overcoated fiber will be compared with a standard PDMS/DVB fiber (i.e. no overcoating) for accuracy, reproducibility and durability. Results obtained from spiked spaghetti sauce samples extracted using the optimized SPME method will then be compared for accuracy, reproducibility and GC/MS background to extraction and cleanup using QuEChERS.

P-4 Simultaneous quantitation and confirmation of about 500 pesticide residues in food extracts using LC-QTOF accurate mass spectrometry

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Fast and comprehensive full scan accurate mass screening and quantitation became an excellent tool in food control in particular if hundreds of pesticides have to be measured in a short time frame. Additionally to the high number of targets the technique takes advantage of unknown evaluation and retrospective analysis. This study evaluates the performance achieved with a high performance QTOF, new software developed for this purpose and a very high quality data base. For the study, a set of 481 pesticides was selected. Matrix based dilution series (10 µg/kg – 2000 µg/kg, QuEChERS-extracts: tomato, summer squash, potato, orange) of the pesticide-mix were analyzed by an UHPLC connected to an impact II QTOF (Bruker Daltonik GmbH) mass spectrometer using a 15 min gradient. Data acquisition is performed in alternating between full scan and bbCID all ion fragmentation mode. Automatic data evaluation is performed using TASQ 1.1 (Bruker Daltonik GmbH), a dedicated software for target analysis screening and quantitation. For automated confident identification we use RT, accurate mass, isotopic pattern and up to 3 qualifier ions in full scan and 7 qualifier ions in bbCID. For tomato extract 454 of 481 pesticides or 94.3 % of the pesticides could be could be identified at 10 µg/kg and 98.9% at 50 µg/kg. For orange extract the numbers were 95.0% at 10 µg/kg and 98.9 at 50 µg/kg; for summer squash extract 91.3% at 10 µg/kg and 95.2% at 50 µg/kg; for potato 98.9% at 10 µg/kg and 99.6% at 50µg/kg.

P-5 Analysis Ultra-Trace Level 1,2,3-Trichloropropane in Water by GC TQMS System in MRM Mode

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1,2,3-Trichloropropane (TCP) is an unregulated chemical without an established Maximum Contaminant Level (MCL). The California notification level is 0.005 micrograms per liter (µg/L) and the Public Health Goal (PHG) has been established at 0.0007µg/L. Two analytical methods developed by California Drinking Water & Radiation Laboratories (CDWRL) are able to detect at 0.005 µg/L level using ion trap Mass Spectrometer (MS) or quadruple MS in SIM mode. The Triple Quadruple Mass Spectrometer (TQMS) in Multiple Reaction Monitoring (MRM) mode is more sensitive and specific and is a choice of technique to meet PHG requirement. The Developed method has excellent sensitivity, linearity and dynamic range were obtained with a limit of detection (LOD) of <0.0002 µg/L, R2 > 0.999, and four orders of dynamic range. Following the liquid-liquid extraction using dichloromethane reagent (100 times enrichment), the Bruker EVOQ GC TQMS system equipped with EI source was used to separate, detect and quantify TCP in water. The MRM ion transitions of m/z 110→75, m/z 110→49 and 112→75 were used as quantifier and qualifier ions, respectively. The transition of m/z 114→75 was used for the stable labeled internal standard. The total run time was 15 minutes.

P-6 A Short Screen and Quantitation LC-MS/MS Method for about 250 Positive and Negative Ion Pesticides in a Single Analysis

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Liquid chromatography coupled with tandem mass spectrometry operated in multiple reaction monitoring (MRM) mode with electrospray ionization (ESI) is widely used for pesticides in food testing. However, two separate runs for positive charged or negative charged pesticides were performed in the common practice, especially when involving hundreds of MRM transitions. A short method was developed for monitoring about 250 positive and negative ion pesticides in 9 min gradient run plus 3 min column re-equilibration time. Two MRM transitions were used for each pesticide, one for quantifier ion and one for qualifier ion. Bruker EVOQ Elite LC TQMS system coupled with Dikma Spursil C18, 3 µm, 50 mm (L) x 3 mm (ID) column was used for separation, detection and quantitation. The Bruker MS system provide fast positive/negative switching allowing for simultaneous determination of positive and negative co-eluting compounds numbering in

the hundreds without any negative performance effects. The preliminary results showed that both positive and negative co-eluting peaks have R^2 >0.99 with linear range 1 to 100 ng/mL. The polar group on the stationary phase of the column provides better retention to polar pesticides and short column with 400 µL/min LC flow provides high throughput separations. AOAC QuEChERS official method was used for sample extraction.

P-7 An Improved Sample Preparation Method for the Analysis of Pesticides in a Challenging Chili Pepper Extract

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Kalsec®'s RM Chili Extract, Okala is a very challenging matrix for pesticide analysis due to the amount of fat and other chili pepper components co-extracted with the capsaicinoids. Our original sample preparation scheme required that the sample be dissolved in acetone, filtered, and directly injected onto the UPLC/MS/MS instrument for analysis (i.e., dilute and shoot). These sample preparations were very concentrated in both lipids and acetone soluble chili pepper matrix components including pigments. The presence of large amounts of matrix components in the injected sample preparations can cause issues with both sensitivity of detection due to ion suppression in the mass spectrometer source and cleanliness of both the injector and the mass spectrometer. Several variations of QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) sample preparation methods were reviewed including methods recommended for high fat samples. The sample preparation method which was found to cause the least amount of ion suppression includes a freeze-out step for the removal of lipids and other matrix components which are sparingly soluble in acetonitrile. This improved sample preparation method addresses both sensitivity and cleanliness concerns.

P-8 Analysis of Pesticide Residues and Mycotoxins in Marijuana using QuEChERS Extraction and ChloroFiltr dSPE Cleanup

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Twenty-four states and Washington, D.C. have passed laws allowing marijuana to be used for medicinal purposes, and in some cases recreationally. However, the use of marijuana continues to be a Federal offense and the Food and Drug Administration has not found any botanical form of marijuana to be safe or effective in treating any medical conditions. Spurred by recent trends in legalization, interest in marijuana and marijuana-based products (e.g. concentrated oils, soda, candy and other edible forms) has dramatically increased. Like any other crop, pesticides are commonly used in marijuana cultivation to protect plants from pests and improve growth yields. However, pesticide residues can pose significant health risks, especially with chronic exposure. The warm, wet conditions ideal for growing cannabis are also conducive to the growth of molds and fungi which are capable of producing carcinogenic mycotoxins, including aflatoxins and ochratoxin A. As a result, testing for the presence of pesticides and mycotoxins in marijuana is essential to ensure consumer safety. Only a few states, including Massachusetts and Nevada, have introduced legislation for the analysis of pesticides and mycotoxins, while other states are in the process of implementing legislation. This poster will outline a QuEChERS based method for the simultaneous analysis of pesticides and mycotoxins in marijuana products. Dispersive-SPE cleanup is carried out using ChloroFiltr, a unique polymeric sorbent for the removal of chlorophyll that unlike graphitized carbon black (GCB) does not result in the loss of planar analytes. UHPLC-MS/MS is used for the analysis of the pesticides and mycotoxins.

P-9 A solid-phase extraction liquid chromatography/tandem mass spectrometry method for analysis of adducts formed between DNA and *epoxide of safrole in vitro and in mice*

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Safrole (1-allyl-3,4-methylenedioxybenzene), the main component of sassafras oil, occurs naturally in a variety of spices and herbs, such as Nutmeg, Basil Rosemary, and black pepper. People are potentially exposed to safrole in their daily life. Safrole-2',3'- oxide (SAFO) is a reactive electrophilic metabolite of the hepatocarcinogen safrole. A previous study using ^{32}P-postlabeling analysis have failed to detect SAFO-DNA adducts in safrole- and SAFO-treated mice. However, our recent data clearly demonstrate that SAFO significantly induced DNA strand breaks and micronuclei formation in SAFO-exposed mice. Moreover, SAFO-induced DNA adducts were detected in mouse urine. In the present study, we aimed to further investigate SAFO-induced DNA adducts in mouse liver and in human HepG2 cells. Using a solid-phase extraction liquid chromatography/tandem mass spectrometry method, N7-(3-benzo[1,3]dioxol-5-yl-2-hydroxypropyl)guanine(N7γ-

SAFO-Gua), N1-(3-benzo[1,3]dioxol-5-yl-2-hydroxypropyl)adenine(N1γ-SAFO-dAdo) were detected in HepG2 cells treated with 250 and 375 µM SAFO. N7γ-SAFO-Gua in liver DNA and urine samples were further analyzed in female CD-1 mice repeatedly treated with safrole (150 and 300 mg/kg/day) and SAFO (30, 60, 90, and 120 mg/kg/day) via ip injection for consecutive 28 days. We observed a dose-dependent increase in N7γ-SAFO-Gua levels in liver and urine. Our results provide the first clear evidence that SAFO indeed causes the formation of DNA adducts in mouse liver. These data not only further confirms the genotoxicity of SAFO, but SAFO-DNA adducts could potentially also serve as cancer risk-associated biomarkers.

P-10 Methodological Advances and Challenges in Long Term Pesticide Monitoring in Lake Erie and Ohio River Basins

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Pesticide use for crop production has increased thousand fold over the past 35 years with introduction of newer pesticides having more complex chemical characteristics. The National Center for Water Quality Research has been monitoring pesticide concentrations in 10 rivers in the Lake Erie and Ohio River basins continuously since 1980. Since then we have measured 20 pesticides that are widely used in agriculture in nearly 20,000 samples. The program has gone through several methodological advancements through the decades, to generate more accurate and high quality data. Our pesticide monitoring program currently focuses on triazines, acetanilides, organophosphates, dinitroanilines and carbamates, because of their high row-crop agricultural use in the local areas. Advancements have been constantly made to develop and maintain cutting edge multi-residue methods. Originally in the 1980s we used separatory funnel extractions and packed column chromatography. Methodological advancements helped us to transition into the modern day solid phase extraction and method development and optimization using GC/MS and LC/MS/MS systems. Significant improvements have been made in lowering the detection limits from >1 ppm to <0.01ppm along with improvements in % recovery range, % RSD and decreasing instrumental run-time. Method development and validation work is currently undergoing with newer classes of pesticides such as 2,4-D, Dicamba and 2,4,5-T in acid herbicide group and Glyphosate and AMPA in organophosphorus group. This presentation will summarize the improvements made in sample extraction, analyses and detection throughout the years. Challenges faced in developing the methods for the newer classes of pesticides will also be discussed.

P-12 Analysis of 647 pesticides (1,929 MRMs) using a high sensitivity LC-MS/MS with a 10.5 minute gradient

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A fast, selective and highly sensitive method has been developed for the quantitation of 647 pesticides using a single LC-MS/MS method with 1,929 transitions and a LC gradient time of only 10.5 minutes. For each target pesticide 3 MRM transitions were utilised to provide additional data confidence in reporting results in comparison to the conventional 2 transitions used in most methods. Acetonitrile QuEChERS extracts were directly injected without the need for dilution prior to injection by using an automated auto-sampler technique. Food extracts of mint, tomato and apple were supplied a commercial laboratory following established QuEChERS protocols. Linearity was assessed over a six point calibration curve from 0.002 - 0.1 mg/kg (2 - 100 pg/µL). All 647 pesticides achieved excellent R^2 values greater than 0.99 in both tomato and mint spiked extracts with typical values greater than 0.996. The high sensitivity MS/MS utilised in this method allowed the dilution of extracts to reduce matrix effects. To assess the robustness of the system and the developed method during routine analysis, repeat injections of a mint matrix sample spiked with 647 pesticides at 0.050 mg/kg were analysed over a 24 hour period. The peak area variance was extremely low over this time period with less than 5.7% RSD for all pesticides measured.

P-13 A multi-residue method for the determination of 114 pesticides in cereals and products of animal origin using the QuEChERs extraction and GC-NCI-MS/MS-TQ detection

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The aim of this study was to develop a GC-Negative Chemical Ionazation (NCI-)MS/MS multi-residue method for the determination of pesticides in difficult and/or fatty matrices, such as cereals, babyfood, products of animal origin, cocoa, coffee, tea, spices and herbs. The GC-MS/MS (TQ-NCI) method is complementary to the GC-MS/MS (TQ-EI) method, in order to reduce false-negative and/or false-positive results for the analysis of complex matrices.

An analytical method combining a QuEChERS extraction with GC-MS/MS (TQ-NCI) detection was developed for the determination of 114 GC-amenable pesticides (including some metabolites/degradation products) in cereals and products of animal origin, with high protein and/or fat content. Various method modifications were tested (e.g. an extra clean up step using partitioning with n-hexane or freezing out, different chemical sorbents, such as PSA and C18 for the dispersive SPE clean up step, extraction time) in order to obtain the highest sensitivity, minimize matrix co-extractives/ interferences and to allow satisfactory long-term chromatography performance.
A Bruker Scion-TQ (using so-called Compound Based Scanning) was used, easily allowing the 385 transitions in the acquisition method (30-min. run time). The scan time for each compound (each with 2 to 5 SRMs) was automatically optimized (from 37 to 225 ms).
Validation of the method was carried out for 114 representative pesticides for wheat, eggs and butter. Recovery studies were performed with spiking levels of 0.005, 0.01 and 0.02 mg/kg for wheat and 0.01, 0.02 and 0.05 mg/kg for products of animal origin. The majority of compounds (85, 86 and 58 %, for wheat, eggs and butter, respectively) met the EU SANTE method validation criteria (i.e. average recoveries in the range 70-120%, with RSD <20%), at the lowest tested spike level. Thus, validated LOQs of 0.005 or 0.01 mg/kg were easily achieved. Only non-polar compounds (28%), at all the spike levels in butter matrix, showed recoveries well below 70 %.
The method has been applied successfully in routine analysis of cereals, products of animal origin and other difficult matrices. A typical example of survey results for vine leaves will be shown.

P-14 Performance evaluation of UHPLC-Q-TOF MS and comparison with UHPLC-TQ-MS/MS and Q-Orbitrap MS for pesticide residues analysis in food samples

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The requirements for the reliable quantification and identification of pesticide residues in fruits, vegetables, cereals and other food products at still decreasing detection levels are continuously evolving with time. Until now, GC-full-scan mass spectrometry or GC-MS/MS and LC-MS/MS Triple quad mass spectrometry at unit resolution are still considered as acceptable for “unequivocal” identification. However, the advancement of MS-technology, especially the introduction of accurate-mass high-resolution mass spectrometry (HRMS) will gradually lift the identification criteria to a higher level. The success of HRMS-instrumentation has been hampered for a long time by limited sensitivity and resolving power, saturation effects due to high levels of coeluting matrix interferences and the lack of user-friendly software for identification and quantification. These disadvantages are rapidly disappearing with the newest generation HRMS-instruments (based on Time-of-Flight or Orbitrap principle) and software.
Our laboratory acquired recently a Bruker UHPLC-Q-TOF HRMS-Impact II instrument, with a resolving power up to 50.000 (at m/z 800). A critical evaluation of the merits of UHPLC-Q-TOF compared to our Waters UHPLC-triple quad MS/MS (XEVO-TQ-S) was performed as to the achievable detection levels and false-positives/false-negatives rates, for various settings of the UHPLC-Q-TOF (mass accuracy error, retention time window, required isotopic ions and/or fragment ions, ion ratios). For these experiments, spiked samples (10, 20 and 50 ug/kg) of a set of representative sample types (lettuce, orange, wheat and tea) were analysed. Results of the comparison will be presented. Also, some preliminary results of a comparison of UHPLC-Q-TOF with a UHPLC-Q-Orbitrap instrument will be shown for a range of standard concentrations in matrix extracts. Initial validation results for the NL-extraction method combined with UHPLC-Q-TOF detection for identification and quantification of a priority pesticides mixture (155 analytes) for lettuce and orange (at spike levels of 10, 20 and 50 ug kg^{-1}) will be presented. Good recoveries, method LOQs, repeatability RSDs , mass accuracy and ion ratio stability were obtained. The method has already successfully been implemented for routine samples series and proficiency tests.

P-15 Simultaneous Analysis of the Amitraz and its Metabolites in Chili Pepper and Mandarin using LC-MS/MS

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The rapid method for simultaneous analysis of insecticide, amitraz and its three metabolites (2,4-dimethylaniline (DMA), 2,4-dimethylformamidine (DMF) and N-2,4-dimethylphenyl-N-methylformamidine (DMPF)) in chili pepper and mandarin by liquid chromatography tandem mass spectrometry (LC-MS/MS, SHIMADZU LCMS-8050™) was developed. The target compounds were analyzed on LC-MS/MS with positive electrospray ionization and selected reaction monitoring. For the best sensitivity in LC-MS/MS, water and methanol with formic acid (0.1 %) were selected for mobile phase. Due to the interference caused by in-source fragmentation of amitraz into DMPF and DMA after passing through the column, complete chromatographic separation of the four compounds was required. The effects of different pH on the QuEChERS extraction of amitraz were studied. The recovery of amitraz was the highest at pH 10 of the extraction solution. To evaluate validity of the final optimized method, recovery tests were performed with untreated chili pepper and mandarin at spiking

levels 1, 10 and 50 times the limits of quantitation (LOQ) (n=3). At all fortification levels, the accuracy and precision results satisfied between 70 and 120% associated with relative standard deviation of ≤10% for amitraz and all the metabolites. Quantitation was performed using matrix matched calibration curves at concentration ranged from 0.005 to 1.0 mg/kg. Correlation coefficients (R^2) of calibration curves was >0.99 for all the target compounds. LOQ of amitraz, DMF and DMPF were 0.01 mg/kg and DMA was 0.02 mg/kg. As a result, the optimized method was proved to be efficient and rapid in the pesticide residue analysis of chili pepper and mandarin.

P-16 Simultaneous Analysis of Pesticides and Ochratoxin A in Red pepper powder using LC-MS/MS and GC-MS/MS

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A large amount—260,000 tons—of red pepper is consumed annually in Korea, where the people prefer hot and pungent to sweet foods. Concern has recently grown among consumers over contamination of red pepper powder by pesticides and mycotoxins. In our previous study, red pepper is highly contaminated by ocrhratoxin A. During production chain of red pepper powder, mycotoxigenic fungi infect Capsicum annuum, crop of red pepper, at pre-harvest stages and generate mycotoxin at pre-harvest stages. In this study, we tried to elucidate the relationship between pesticide use and ochratoxin A occurrence in red pepper powder. Capsicum annuum is chosen as proper model organism for this purpose, since occurrence frequency of ochratoxin A and pesticide residue is high enough to evaluate statistically the efficacy of pesticide as prevention measure against mycotoxin occurrence.
Nowadays, generic extraction approaches such as QuEChERS in combination with tandem mass spectrometry enable to analyze wide array of pesticides and mycotoxins simultaneously providing a clear clues on the post mortem evidence of pesticide use history and mycotoxin occurrence. We developed and validated the method for simultaneous analysis of 320 pesticides and ochratoxin A, which met criteria of validation parameters such as trueness, precision, and linearity upon DG SANCO(12571/2014) for most of analytes and also suited to MRL upon Korean food code.
Our statistical investigation showed little correlation between mycotoxin occurrence and fungicides residues contradictory to laboratory experiment result carried out by other research groups. Insecticides which control insect, carrier of ochratoxigenic fungi was found to have a littlle effects on reduction of ochratoxin A.

P-17 Survey of Plant Growth Regulators in Consumer Fertilizer Products using LC-MS/MS

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In Canada, plant growth regulators (PGRs) are regulated under the Fertilizers Act or the Pesticide Control Products Act, depending on their specific mode of action. To date, there's been little monitoring of PGRs in commercial fertilizer products. In order to determine if unregistered PGRs are added to commercial products, a limited and informal survey was initiated. Six fertilizer products were purchased, off-the-shelf, in British Columbia and analyzed for 10 common PGRs. The method was a simple dilution and subsequent analysis by LC-MS/MS. The survey showed the presence of three unregistered PGRs in three of the six products: daminozide, ethephon, and paclobutrazol. This rather surprising finding may lead to an expanded and more formal monitoring program for unregistered PGRs in commercial fertilizer products in Canada.

P-18 Determination of 234 Pesticides in Juices using UHPLC/ESI-MS/MS and QuEChERS

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This poster presents an UHPLC/ESI-MS/MS method to determine 234 pesticides in juices. Pesticide residues were extracted from juice samples using a procedure known as QuEChERS (quick, easy, cheap, effective, rugged and safe). UHPLC/ESI-MS/MS quantification was achieved using matrix-matched standard calibration curves with isotopically labelled standards or a chemical analogue as internal standards. The calibration curves consisted of six points (5, 25, 100, 200, 300 and 500 µg/L as in sample) and the method was validated at four concentration levels (10, 90, 240 and 400 µg/L) in triplicate with a total of eight different fruit and vegetable juice matrices on two separate days per matrix. The method performance parameters that included overall recovery, intermediate precision and measurement uncertainty were evaluated according to a nested experimental design. Approximately, 97% of the spiked pesticides in fruit juices and 96% in vegetable juices had recoveries between 71 and 120%; 97% of the spiked pesticides in both fruit and vegetable juices had intermediate precision ≤ 20%; and 97% of the spiked pesticides in fruit and vegetable juices showed measurement uncertainty ≤ 50%. From a pilot study of 50 juice samples, 12 samples were found positive with pesticide residues ranging from 6 to 227µg/L. The incurred samples contained a total of 17 different pesticides. However, no incurred residues in the samples exceeded Canadian MRLs as expressed in fruits or vegetables.

P-19 Comparative Validation Study of Microflow-LC and UHPLC with Orbitrap MS for Quantitative Analysis of Pesticides in Food Matrices

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As FDA continues to pursue advancements in pesticide residue analysis, emerging analytical technologies must be evaluated. One such separation technology, microflow liquid chromatography (MFLC), uses a smaller column and lower flow rate than standard LC analysis, and can be installed as an upgraded front-end to existing MS instrumentation. Compared to conventional UHPLC, MFLC is believed to offer greater sensitivity, a substantial reduction in matrix-effect issues and ancillary benefits of reduced solvent usage. This yields savings for operational costs in solvents, reduced waste disposal and resultant positive environmental impact. In the current study, a full method validation was performed for both MFLC and UHPLC front-ends, each paired with a Q-Orbitrap mass spectrometer. Comparisons between the systems were made using both incurred-residue sample analysis and a spiking study on a variety of sample matrices. The two systems were compared with respect to linearity, reproducibility, spike recovery, matrix effects, and limits of detection and quantitation. By comparing the MFLC system to existing UHPLC technology side-by-side, the feasibility of incorporating MFLC for future regulatory analysis can be evaluated.

P-20 High resolution/high mass accuracy MS/MS Libraries for Chemical Residues and Contaminants

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Liquid chromatography-tandem mass spectrometry (LC-MS/MS) is the major tool for analyzing residues and contaminants, such as pesticides, mycotoxins, veterinary drugs, and other emerging chemicals, in complex food matrices. Quantitation and identification of these residues and contaminants require the selection of characteristic precursor and product ions. Typically, structural elucidation of product ions is not determined but it would be helpful to have this information to ensure accurate identification of the chemical to further support regulatory monitoring programs. LC-HRMS data and spectra of pesticides, mycotoxins, and veterinary drugs were acquired using a Q-Orbital trap mass analyzer in positive electrospray ionization mode and full scan and dd-MS/MS modes. By applying mass fragmentation software and data from the literature to high resolution/high mass accuracy MS/MS spectra, rational structures of product ions were generated for over 600 pesticides, 55 mycotoxins, and 100 veterinary drugs. These high resolution/high mass accurate full scan MS and MS/MS libraries can be used to generate precursor and product ions for a compound database enabling software algorithms to automatically screen food matrices for residues and contaminants. These MS/MS libraries will help researchers select appropriate MS/MS product ion transitions for low resolution mass spectrometry. Finally, we demonstrate how high quality MS/MS spectra can support, as well as increase, our understanding of the fragmentation mechanism of an ergot mycotoxin (ergocornine) from previously published low resolution MS/MS results.

P-21 Investigation of Free and Reversibly Bound Sulfite using a Non-Targeted High-Resolution Mass Spectrometry Approach

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Sulfites are food additives used to limit browning and microbial growth. Sensitive individuals have reported severe allergic-type reactions following consumption of sulfite treated foods. In 1986, the US FDA mandated that sulfites be declared on the label of any product containing in excess of 10 ppm SO_2. The current sulfite regulatory method, the optimized Monier-Williams (MW), is a distillation method where the sample is refluxed with a hydrochloric solution to release sulfite from the food matrix. The sulfur dioxide gas is then bubbled through hydrogen peroxide to form sulfuric acid which can be titrated with sodium hydroxide. While this method is reliable for most matrices, it is time consuming and requires specialized glassware. Recently, an improved LC-MS/MS method was developed that converts sulfite to the stable formaldehyde adduct, hydroxymethylsulfonate, through a simple extraction with a dilute formaldehyde solution. In direct comparisons of these two methods, significant differences in the results were observed for some of the food

matrices in select cases. This discrepancy could potentially be due to differences in the release of bound sulfite from the matrices since the extraction techniques of the method vary. Non-targeted screening using high-resolution mass spectrometry (HRMS) of sulfited and non-sulfited apricots was conducted in order to identify the main food components to which the sulfite was binding. Additional matrices were investigated to determine if differences in the sulfite bound food components could account for the differences observed in the two matrices.

P-22 A Simple and Effective Cleanup Procedure to Produce QuEChERS Extracts That Are More Compatible with Gas Chromatography

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A simple and effective clean up procedure has been developed to make QuEChERS extracts much more compatible with gas chromatography (GC). Without additional cleanup procedures, QuEChERS extracts of high chlorophyll sample matrices are known to degrade the GC performance of several pesticides, including captan, folpet, methoxychlor and DDT. The described procedure, which does not require GCB or toluene, reduces most of these problems with minimal loss of planar nonpolar compounds like hexachlorobenzene (≥ 80% recovery through the cleanup steps). The described method simply adds water back to the QuEChERS extract (prior to dSPE cleanup), vortexes the solution and filters through a 0.2 µ PTFE filter. The percent of water used was optimized to remove chlorophyll, but maintain adequate recovery of hexachlorobenzene and other nonpolar pesticides. After filtration, water is removed using the same QuEChERS saltout approach. Additional dSPE cleanup and drying is performed using MgSO4/PSA/Chlorofiltr®. Improvement in chromatographic reproducibility was also observed using this procedure on fatty matrices like animal feeds and olive oil. Details of the described procedure and validation results are discussed.

P-23 Determination of Pesticides in High Fat Food Products using a Modification of the Irvine Rapid Analytical Method (IRAM) with LC-MS/MS

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The IRAM method (FDA LIB 4495), previously validated for high moisture food products, was extended to high fat food products. Minor modifications were made – lower sample weight and higher extraction solvent volume. The method was validated on the LC-MS/MS platform with three matrices, almond, olive oil and fish (tilapia), fortified at 10 ppb, 50 ppb and 100 ppb. The recovery data are acceptable within the range expected for residue analysis by FDA Pesticides Analysis Program guidelines and common industry standards (AOAC, Codex).
The average recoveries LC/MS/MS for 200 pesticides fortified at 10 ppb, 50 ppb, and 100 ppb were 90% with RSD < 5% for 90% of the analytes studied. The proposed method has demonstrated it is reliable and dependable for extraction and detection of pesticide residues in fatty food commodities.

P-24 A QuEChERS Sample Extraction Procedure Modified with Enhanced Matrix Removal Sorbent for Determination of Pesticide Residues in Edible Oil

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The extraction of pesticide and chemical residues from high lipid food matrices requires a balance between the efficiency of the extraction technique and the cleanness of the final solution. The extraction process is more challenging than with other food matrices because of the difficulty obtaining phase separation between the extracting organic solvent and oil layers. The oil is miscible in organic solvent thus requiring a special technique to minimize peak interference during the instrumentation elution and detection process. A newly developed enhanced matrix removal (EMR) sorbent selectively adsorbs the lipids; the chromatography improves with this minimization of lipids in the sample extract.
The proposed method was optimized by the mixture of extraction solvents and ratio of water added during sample extraction. The resulting extraction solvent is a 50/50 acetonitrile/acetone mixture, which gives the most acceptable extraction efficiency and recovery of target compounds. The method detection limit on LC-MS/MS and GC-MS/MS platforms are 10 ppb or lower for more than 90% of about 400 Accustandard mix. The recovery, linearity and method detection limit are reported and acceptable.
The proposed method is more environmental friendly because it does not use toluene or ethyl acetate in the extraction process.

P-25 Sensitive and Fast Analysis of Glyphosate, Glufosinate and AMPA in Food Matrices with HILIC HPLC and Mass Spectrometry

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Glyphosate is the world's bestselling herbicide and the most widely used herbicide in the United States as well as in the world. Studies show that glyphosate has serious toxic effects on health and the environment. The extraction of glyphosate, glufosinate and AMPA is a challenge because of the high polarity of these compounds, and broad screening methods do not adequately extract these compounds from food matrices. An HPLC or LC-MS/MS method for general screening usually could not be used to analyze these three compounds either. Usually, specialized extraction coupled with modified HPLC detection methods are used in the laboratories, but these methods are not always efficient, sensitive, or practical.
Therefore, a faster, more sensitive and high-throughput method is desired. The proposed method uses a new HPLC gradient program with a HILIC column and HILIC mode conditions. Based on the robustness and sensitivity of this HILIC method, the sample extraction method of FDA LIB 4595 and LIB 4596 were modified for simplicity and method harmonization.
Data published by the EU based on ion-exchange HPLC was compared to data from the proposed HILIC method which showed that the HILIC method is simpler, faster, more sensitive, and convenient.

P-26 Differential Mobility Spectrometry for the Analysis of Stilbene Hormone Residues in Seafood

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Differences in the collisional cross-section (size, shape) and charge state of gas-phase ions affect how they move in an electric field. In chemical residue analysis, enhanced selectivity can be achieved by coupling ion mobility spectrometry with mass spectrometry. Differential mobility spectrometry (DMS) provides an orthogonal analytical tool for the separation of isobaric compounds, either to distinguish two analytes of interest or to isolate an analyte from matrix components. In the current study, we applied LC-DMS-MS/MS to the quantitative analysis of diethylstilbestrol, dienestrol, and hexestrol, three non-steroidal synthetic estrogenic compounds belonging to the stilbene class of compounds. Stilbenes have been banned from use in food animal production because of their potential to cause cancer and birth defects in humans. In a previously developed LC-MS/MS method for stilbene residue analysis in fish, matrix interferences were noted in fatty salmon and catfish samples. The inclusion of ion separation based on DMS significantly reduced the background signal from the fish matrix resulting in improved signal-to-noise ratios for the stilbene residues. Solvent modifiers with different properties were investigated to increase clustering and further enhance mobility differences between stilbene molecules and matrix components. The overall analytical performance of the LC-MS/MS and LC-DMS-MS/MS methods was compared in this study.

P-27 Optimization of a Cleanup and Extraction Procedure for the LC/MS analysis of Veterinary Drugs in Fish and Shrimp

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A rapid, wide-ranging fish and shrimp extraction method is described. Although a large number of cleanup strategies for veterinary drug residue LC/MS methods are available in the literature, recently specific lipid cleanup technologies have been introduced. Removal of lipids is especially important for high fat-containing matrices such as salmon and other fish. This procedure uses a new type of solid phase extraction (SPE) cartridge named Oasis® PRiME HLB to remove interfering matrix components from tissue extracts. The different aspects to optimizing the extraction and cleanup procedure for fish and shrimp are discussed. The final method extracts tissue by vortex mixing with acetonitrile which has been acidified with 2% acetic acid and 0.2% r-toluenesulfonic acid. A centrifuged portion of the extract is passed through the SPE cartridge, evaporated, reconstituted and injected into a Thermo Q-Exactive high resolution Orbitrap mass spectrometer for analysis. The acidified acetonitrile was the best overall choice for extracting both non-polar and polar veterinary drugs in tilapia, catfish, salmon and shrimp. The compounds tested included sulfonamides, tetracyclines, β-lactams, macrolides, avermectins, triphenylmethane dyes, fluoroquinolones, quinolones, nitroimadazoles, and benzimidazoles.

P-28 Development and validation of a LC/MS/MS method for the determination of isoeugenol in finfish

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A simple, fast, selective and sensitive LC/MS method was developed and validated for the determination of isoeugenol in finfish using an off-line dansyl chloride derivatization step to enhance signal intensity. Eugenol was used as an internal standard. The sample was extracted with 10 mL acetone using a Gino-Grinder, followed by derivatization with dansyl chloride and analysis by multiple reaction monitoring liquid chromatography electrospray mass spectrometry. The LC separation was achieved using a Zorbax Eclipse XDB-C18, 4.6x50 mm, 1.8 µ analytical column combined with an isocratic mobile phase at a flow rate of 500 µL/min. The method detection limit ranged from 0.2 – 0.71 ng/g in six fish species. The limit of quantification was 2.5 ng/g in each fish species. Recoveries of the isoeugenol from the different fish at three different levels over two different days ranged from 91.2% to 108.0%. Precision ($\%RSD_R$) values determined at 10 ppb were 4.3%, 8.0%, 5.3%, 5.1%, 2.6% and 8.0% for tilapia, catfish, trout, salmon, hybrid striped bass and yellow perch, respectively. Linearity was studied in the range 2.5 – 40 ng/g, and the obtained determination coefficients (R^2) were above 0.997 for all matrices.

P-29 Development and validation of a direct determination of glyphosate, glufosinate, and aminomethylphosphonic acid in honey by liquid chromatography/tandem mass spectrometry

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A simple high-throughput liquid chromatography/tandem mass spectrometry (LC-MS/MS) method was developed for the determination of glyphosate, glufosinate, and aminomethylphosphonic acid (AMPA) in honey using a reversed-phase column with weak anion-exchange and cation-exchange mixed-mode (Acclaim™ Trinity™ Q1). One gram of sample was shaken with water containing ethylenediaminetetraacetic acid disodium salt (Na_2EDTA) and acetic acid for five minutes. After centrifugation, the supernatant was mixed with internal standard and directly injected and analyzed in ten minutes by LC-MS/MS with no sample concentration or derivatization steps. Two precursor/product ion transitions were monitored in the method for each target compound to achieve true positive identification. Three internal standards corresponding to each analyte were used to correct for matrix suppression effects and/or instrument signal drift. The linearity of the detector response was demonstrated in the range of 2.5 to 250 ng/mL for each analyte with a coefficient of determination (R^2) value of 0.998. Through the use of this internal standard calibration method, the average recovery for all analytes at 25, 50, 100, and 500 ng/g (n = 11) ranged from 87 to 111% with a relative standard deviation of less than 12%.

P-30 Rapid, new, methods for the analysis of 3-MCPD and 1,3 DCP in soy sauce

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Acid hydrolyzed vegetable protein (aHVP) is used for flavoring a wide variety of foods and also in the production of non-fermented soy sauce. During the production of aHVP, chloropropanols including 3-monochloropropane-1,2-diol (3-MCPD) and 1,3 dichloropropane-2-ol (1,3 DCP) can be formed through the reaction of the hydrochloric acid catalyst and residual fat and the reaction of 3-MCPD with acetic acid, respectively. 3-MCPD is a carcinogen and a suspected genotoxin in humans. The European Union (EU) has set a maximum level of 0.02 ppm of 3-MCPD in aHVP, and the Food and Drug Administration (FDA) set a guidance limit of 1 ppm of 3-MCPD in aHVP. Prior to the guidance level being set, a survey of 55 samples performed by the FDA found 33% of samples to have concentrations greater than 1 ppm. An AOAC method was used for this analysis, which is time consuming, labor intensive, and requires excessive solvents. A new survey of 60 sauces was performed in 2015 to determine if concentrations have changed since 2008 using newer, more rapid methods. An alternative method was investigated that uses phenylboronic acid as a derivatizing agent. Additionally, a new technique using microvial thermal desorption coupled with GCMS was developed involving minimal sample preparation and the rapid assessment of 3-MCPD and 1,3 DCP contamination in soy sauce samples.

P-31 Acceptance Criteria for Confirmation of Identity of Chemical Residues using Exact Mass Data within US FDA Office of Foods and Veterinary Medicine

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The Office of Foods and Veterinary Medicine of the US Food and Drug Administration has developed acceptance criteria for the confirmation of identity of chemical residues using exact mass data collected with high resolution mass spectrometry (HRMS). With recent technical advances in HRMS and its increased use in the analysis of foods and veterinary medicines, it was important to develop guidance so that users of HRMS are consistent in evaluating and comparing results for regulatory use. These criteria are meant to supplement the 2002 FDA Center for Veterinary Medicine (CVM) published Guidance for Industry (#118) titled "Mass Spectrometry for Confirmation of the Identity of Animal Drug Residues". The primary focus of this document is for the use of HRMS for targeted analysis when a comparison standard is available, although aspects of non-targeted analysis are discussed. The criteria addressed include requirements for signal intensity, retention time matching, number of structurally significant ions, and mass accuracy for precursor and product ions. The guidance has been approved and is now available at: http://www.fda.gov/ScienceResearch/FieldScience/ucm273423.htm

P-32 Rapid Detection of Amantadine in Poultry Samples

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Amantadine was approved for use both as an antiviral and antiparkinsonian drug by US Food and Drug Administration respectively in 1960s and 1970s. Due to gained resistance by the flu virus, it is no longer recommended for treatment of influenza in the United States in 2009 by the US Centers for Disease Control and Prevention. Additionally, its effectiveness as an antiparkinsonian drug is also undermined, with a 2003 Cochrane Review concluding that there was insufficient evidence in support or against its efficacy and safety. In 2005, Chinese poultry farmers were reported to have used amantadine to protect birds against avian influenza. Thus it has since been banned for use as veterinary drug by Chinese government regulatory agencies. To strictly enforce this regulation, a sensitive and high throughput screen method is needed for poultry samples. We have developed a rapid and sensitive method to meet this requirement. Our new method uses an enzyme-linked immunoassay technology to detect amantadine, which is based on selective antibody-antigen binding interaction. A simple, rapid, sensitive, and robust sample preparation protocol for poultry samples was developed, allowing a very sensitive detection limit at 0.25 ppb. Compared to LC-MS based assay, our assay offers a more sensitive detection limit at a much lower cost and a much higher throughput capability. Our assay is a very important tool to assure food safety and quality free of amantadine.

P-33 A Risk-Benefit Analysis for Commonly Consumed Finfish: DHA and EPA intake versus Cadmium, Lead or Mercury Exposure

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Seafood is nutritionally beneficial. It is the dominant natural source of the essential long-chain omega-3 fats, DHA and EPA in the human diet, is high in protein and is low in saturated fat. According to recent studies however, 85% of Americans do not achieve the USDA recommended consumption rate of seafood based on omega-3 fatty acid intake of two eight ounce portions per week. Seafood and more specifically finfish may contain environmental contaminants including the heavy metals; cadmium, lead and mercury. All finfish contain methylmercury, a neurotoxin that readily crosses the blood-brain and blood-placental barriers. Typically the higher the trophic level of the fish species the higher the mercury levels. Farmed fish however are known to have relatively low levels of mercury and a recent survey indicates that finfish fish are also low in cadmium and lead. In this presentation we will focus on popularly consumed species, tuna (fresh, canned and sushi grade), swordfish, mahi mahi, red snapper, red grouper and salmon, and evaluate the best choice to for the consumer to make to reduce the risk of heavy metal exposure and maximize the consumption of quality fats and protein.

P-34 Florida Department of Agriculture Chemical Residue Annual Report FY 15-16

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The Bureau of Chemical Residue laboratories screened between 317 and 500 pesticides in fresh fruits and vegetables during the fiscal year from July 1, 2015 to June 30, 2016. During that time about 1,300 samples, representing over 90 different commodities were analyzed in the pesticide residue tolerance enforcement program, and approximately 30 violations were reported and follow-up investigations conducted. In addition approximately 1,300 samples were analyzed for the Pesticide Data Program. In support of research to combat citrus greening, the Bureau also developed a method for the analysis of penicillin and two metabolites in citrus and analyzed over 100 samples. Over 750,000 analyses were conducted. This poster will summarize the functions of the Bureau laboratories and the major findings for FY 15-16.

P-35 How repeatable is your laboratory's Sample Processing? An Estimation of Measurement Uncertainty including Laboratory Sample Processing

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Laboratories frequently estimate their measurement uncertainty based on an analytical method's quality control (QC) spike repeatability. This poster presents the Florida Department of Agriculture, Chemical Residue Laboratories (CRL) quality control procedure to estimate measurement uncertainty which includes sample processing (e.g. room temperature and cryogenic blending) and residue analysis. The CRL receives and processes over 3,000 samples annually for pesticide residue analysis. Samples include fresh fruits, vegetables and honey for regulatory enforcement of maximum residue limits (MRLs). If a sample is found violative, regulatory actions could result in destruction of valuable food product. Tremendous resources are spent on the development and validation of new analytical instrumentation and extraction methods for chemical residues, but the important step of sample processing may be overlooked. As every matrix is different, it is important to ensure all parts of the sample handling are checked including cleaning equipment; chopping, blending and storage of samples. The laboratory participates in over six proficiency test samples annually which checks the extraction and analysis of samples, but this does not check sample processing as proficiency test samples do not require processing. This presentation focuses on the laboratory's ability to reproduce results for samples with incurred residues by repeat analysis from the comminuted portion. Results from initial repeatability studies will be presented. It is hoped that, over time, many matrix / analyte combinations will be screened and we will have a better understanding of the uncertainty in laboratory processing.

P-36 Identification of Penicillin G and its Metabolites in Citrus Fruits Affected by Huanglongbing using UHPLC-MS/MS

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We developed and validated a method for the extraction, identification and quantitation of penicillin G and its metabolites in a variety of citrus fruits by employing sequential liquid/liquid and solid-phase extraction techniques in conjunction with UHPLC-MS/MS. Two product ion transitions per analyte were required for identification which contributes to a high degree of selectivity. The limit of detection (LOD) of penicillin G and its metabolites was found to be 0.25 ng/g when 2 g of citrus were extracted. This method allowed determining residue levels of penicillin G and metabolites in citrus trees infected with Huanglongbing bacteria or citrus greening disease after antibiotic treatment. We also investigated the stability of penicillin G using two UHPLC-MS/MS systems with variable capabilities (*i.e.* Thermo Q Exactive Orbitrap and Sciex 6500 Qtrap) in various conditions in order to understand its dissipation under field conditions. The findings of this study will provide insight regarding penicillin G residues for food safety purposes, in agricultural and biological applications.

P-37 Rounding up Glyphosate: Method development for glyphosate and AMPA in fruits and vegetables

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Since the introduction of Roundup in the 1970s, the herbicide has enjoyed a long period of use due to its effectiveness and relatively low toxicity. Recently, public outcry has prompted a reassessment of the health impacts of glyphosate, the active ingredient in Roundup. The ubiquitousness of glyphosate in consumer products, specifically residues of glyphosate in food products, poses a problem for monitoring and regulatory agencies without a straightforward method to test for glyphosate and its metabolites. The Florida Department of Agriculture and Consumer Services Chemical Residue Laboratories in the Division of Food Safety have developed a single residue method for the detection and analysis of glyphosate and its main metabolite, AMPA, for routine analysis of fresh fruits and vegetables. The method is based on a solid-liquid extraction of homogenized raw agricultural commodities with water, centrifugation, filtration and SPE clean-up. The pH of the final extracts are adjusted with 50mM ammonium formate buffer (pH 3) and analyzed by LC-MS/MS without derivatization. Retention and peak shape were evaluated on three columns, HILIC, ZIC-HILIC and mixed-mode. Analysis by linear ion trap mass spectrometry was used to maximize sensitivity of the major product ions of glyphosate and AMPA. In addition to standards in matrix, isotopically-labeled internal standards were used to compensate for matrix and process interferences. This poster summarizes a simple and efficient method that allows for the quick analysis of glyphosate an AMPA.

P-38 "The Real World of Standards" Standard Stability of Pesticides and Antibiotics used for Multi-residue Methods

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Standards (i.e. reference materials) are crucial components for sample analysis in a high throughput production laboratory and assuring their integrity is of vital importance. This poster presents the Florida Department of Agriculture, Chemical Residue Laboratories (CRL) standard stability studies for pesticides and antibiotics used for multi-residue methods. The CRL must have the capability to detect and accurately quantify hundreds of chemicals in samples including fresh fruits, vegetables, and honey. If a sample is found violative, regulatory actions could result in destruction of valuable food product, therefore it is imperative standards used for analysis are without question. CRL uses multi-residue compound mixes whenever appropriate to analyze samples in a reasonable amount of time. Because the multi-residue compound mixes are expensive and take a long time to prepare, the laboratory works in prolonging standards life span by checking their stability over a period of 12 months. Preparation, solvent, concentration, and storage are all factors that can influence standards integrity. This presentation will focus on the real world testing and data that the CRL used to determine the stability of pesticides and antibiotics used for multi-residue analysis of samples.

P-39 Investigation of Different Isomeric Forms of Zearalenone (ZEN) Hydroxylated Metabolites by High Resolution Mass Spectrometry Coupled with Ion Mobility that form a single chromatographic peak with an LCMSMS screening method.

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Mycotoxins are natural contaminants that can appear in the feed and food chain as a result of fungal infestation and pose food safety concerns and health issues in animal production or humans. These compounds can exert carcinogenic, estrogenic and immunomodulatory effects, and so are strictly regulated in food and feed. The extreme chemical diversity of mycotoxins and their metabolites produced by plants, microorganism and the animal host can result in a multitude of chemical structures that can be expected from an extract, making screening difficult and prone to errors. Whilst MS techniques are allowing precise determination of the distribution and incidence of some mycotoxins in feeds, these technological advancements become limiting when tracking their metabolites in animal organisms. Furthermore, different levels of toxicity can exist for isomers that cannot be separated by traditional LCMSMS methods.
We demonstrate how separation of isomeric species is possible using a HRMS coupled with ion mobility. We are able to investigate the fragmentation patterns and relative levels of zearalenone (ZEN) hydroxylated metabolites that are generally found as isomeric forms in a single injection.
Zearalenone, α-zearalenol[α-ZOL], β-zearalenol[β-ZOL], α-zearalanol [zeranol, α-ZAL], β-zearalanol [teranol, β-ZAL] and zearalanone [ZAN] can be determined with a UPLCMSMS method using a binary mobile phase of water:methanol containing 0.1% formic. Dried sample extract are generally reconstituted in water/ACN/formic acid in 10mM ammonium acetate loading buffer. However, identification and precise quantification are prone to imprecision when those occur in mixtures due to overlapping chromatographic retention time and common product ions following MRM experiments.

P-40 Simple and Effective Cleanup for UPLC-MS/MS Determination of Veterinary Drug Residues in Egg

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In order to insure public health and safety, reliable analytical methods are necessary to determine veterinary drug residue levels in animal products such as meat, milk and eggs. In order to maximize throughput and minimize costs it is desirable to determine the widest possible range of veterinary drug residues in tissue samples with a single analytical method. This poster presents sample extraction, cleanup and analysis protocols for the multiresidue UPLC-MS/MS determination of veterinary drug residues in egg. Eggs contain significant amounts of protein, fat and lecithin (phospholipid). The protein is removed in the initial solvent extraction step by precipitation and centrifugation. However, significant amounts of fat and phospholipids are co-extracted along with the target veterinary drugs. The presence of these co-extracted substances can lead to interference in the LC-MS analysis, contamination of the analytical column and other components of the UPLC system, and contamination of the mass spectrometer itself. To avoid these complications, the supernatant extract is subjected to a rapid and simple one-step cleanup using an Oasis PRiME HLB cartridge to effectively remove fats and phospholipids. Recovery and precision data are presented for multiple antibiotic residues including tetracyclines, macrolides, amphenicols, quinolones and B-lactams.

P-41 Use of DART-QDa for the Rapid Authentication of Food and Dietary Supplements

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Food and dietary supplement fraud is a closely monitored area. In order to protect consumers, samples need to be rapidly screened to determine the product authenticity. Cinnamon analysis was performed on both cinnamon sticks and the ground spice. Ground cinnamon analysis utilized the QuickStrip module for sample introduction. Omega fatty acid analysis of oil supplements was performed on oil samples diluted in toluene. The DART QuickStrip module was utilized for analysis of the diluted oils using 5 µl sampling spots. Analysis temperature was optimized for each application. Cinnamon analysis was performed at a temperature of 450°C, while fatty acid analysis was performed at a temperature of 200°C. Cinnamon species was able to be determined using the DART-QDa analysis based on the ratio of cinnamaldehyde (m/z 133) to coumarin (m/z 147) in the mass spectrum. DART-QDa results, from both cinnamon sticks and ground cinnamon, demonstrate the difference in cinnamaldehyde and coumarin levels dependent on species. Fatty acid analysis focused on the levels of omega 3, omega 6, and omega 9 fatty acids typically present in commonly consumed dietary oil supplements. Three different dietary oil supplements (fish, flax seed, and safflower oil) were analyzed using the DART-QDa method to authenticate the contents of the supplements as indicated on the bottles. The experimentally determined percentage of fatty acid composition of each supplement matched closely to the percentages disclosed on the supplement bottles. This method can also distinguish source of the oil (fish vs plant) dependent upon the fatty acids identified in each sample.

P-42 Accessible and Efficient Screening of Multiclass Contaminants in Food

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With growing global trade and varying international regulations, the need for multi analyte screening procedures to efficiently detect violating residues is ever increasing. Routine testing laboratories continue to strive for efficient and reliable sample throughput methodologies, where generic analytical conditions are essential. SPE extracts of foodstuffs were run on UPLC separation using a BEH C18 (1.7µm, 2.1x100 mm) analytical column. Targeted multi reaction monitoring methods were used to identify and quantify multiple compounds in a single method. This work reports the development of a screening method for the determination of multiclass multiresidue contaminants in complex foodstuffs, including foods of animal origin. A generic and simplified sample extraction protocol was used with liquid-liquid extraction, followed by a generic reverse phase, solid phase extraction (SPE) procedure. All solvent standards, matrix matched calibration curves and sample extracts were analysed using UPLC coupled with a high sensitivity tandem quadrupole mass spectrometer.
The benefit of the high sensitivity instrument was evaluated, in order to investigate the effect of matrix dilution to overcome common analytical challenges such as ion suppression. Further enhancements in analytical efficiencies were achieved utilizing the functionality of the novel multimode ionisation source, operated at atmospheric pressure. Exploiting the key functionality of the high velocity droplet stream extends the scope of multiclass analytes screened to excelled levels of detection in a single workflow.

P-43 Analysis of LC and GC Amenable Contaminants in Food on a single MS platform

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A suite of pesticides that are both GC and LC amenable were analyzed on a single system. Data was acquired using alternating high and low collision energy states across the full analytical mass range, such that product ions were also generated. The data was screened against a known library of compounds which was automatically interrogated using mass accuracy, isotopic fit and fragment matching. The data was also interrogated for unknown contaminants present and elucidation of the unknowns was determined with the software. The GC and LC data was be handled on a single data analysis platform. Fruit and vegetable samples were spike with GC and LC amenable pesticides at regulatory reporting levels. The samples were extracted using QuEChERS methodology and run on the system in both APGC and ESI mode. The samples were run in a data independent analysis where low- and high-energy data was collected simultaneously giving the product ions of all precursors. The samples were screened against a scientific library in order to confirm the presence of targeted compounds. The confirmation of these compounds was performed using mass accuracy, isotopic fidelity and fragment ion confirmation. The linearity and reproducibility of the system will also be accessed in future work. As well as screening for targeted compounds that are contained within the scientific library, unknown contaminants were also investigated. This was done by comparing blank extracts against spiked samples to determine the differences. The identification of the unknowns was performed by using elemental composition, database searching and fragment identification.

P-44 Environmental Screening of Water Samples Utilizing Ion Mobility Enabled High Resolution Mass Spectrometry

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Companies and environmental regulatory authorities are under pressure to develop screening methods capable of detecting a broad spectrum of environmental contaminants in a single analytical run. Many are turning to High Resolution Mass Spectrometry (HRMS) as part of the solution. Data collected using data independent acquisition of both high and low energy simultaneously with ion mobility separation was interrogated for a range of environmental contaminants, including legacy and emerging polyfluorinated compounds. The same instrument was operated in this mode coupled with a variety of chromatographic techniques and ionizing methods (including ESI and API). By utilizing the different chromatographic and ionization techniques the range of compounds covered can be expanded. Using a fully integrated scientific information system, which performs data processing via Apex 3D peak picking and componentization, a target list of compounds was screened against. In order to identify target compounds against a library mass error, isotopic fidelity and fragment matching was used. A unique measurement that is made via the mobility separation collisional cross section (CCS) giving an extra point of confirmation for known compounds. The mobility separation also allowed the spectral clean up in data allowing identification to be performed with more confidence. The same data was then used to isolate non-targeted compounds by using an array of comparison and discovery tools without the need to reprocess. Identification of these significant compounds of interest is also addressed by discussing elucidating techniques, such as a novel batch elucidation tool, available within the integrated scientific information system.

P-45 Discrimination of Honey of Different Botanical Origins using an Untargeted High-Definition Metabolomic Workflow

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The study demonstrated successful employment of an untargeted high-definition metabolomic approach for floral classification of honey. Initial investigations were undertaken with 5 commercial honeys of polyfloral origin to ascertain whether they could be differentiated by IMS coupled non targeted metabolomics. Once a success approach was confirmed, the experiment was expanded to unifloral honeys of authentic verified origins. Floral origins of the honeys studied were rape, heather, manuka and buckwheat. Honeys were collected over multiple years from 2009-2014 from multiple countries including Lithuania, Poland, Denmark, New Zealand and Norway. UPLC-HDMSE analysis of the samples, including honey pools for QC purposes, was completed in triplicate in both positive and negative ESI in a randomised fashion. Resulting data were processed in Progenesis QI. Unsupervised PCA showed a clear differentiation between the unifloral honeys with Rape and Buckwheat showing the closest association - clearly separated from Manuka and Heather. Further investigations were made with OPLS-DA analysis to elucidate the components responsible for the

differences. Unique markers of all of the honeys were identified - including those consistent with previous literature e.g. methyl syringate, leptosin and leptosperin from Manuka samples. Validation of selected identified markers was undertaken using targeted metabolomics on a UPLC- Xevo TQ-S. Evaluation of the markers of botanical origin discovered in the non-targeted metabolomic approach were compared to those found using a similar experimental workflow using rapid evaporative ionisation MS (REIMS) . The in depth mining metabolomic approach combined with direct sampling at "point of entry" may facilitate authentication techniques at source.

P-46 Rapid Evaporative Ionisation Mass Spectrometry (REIMS) for Food Authenticity Testing

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Due to their high market value, meat and fish products are often targets for species substitution, adulteration, or mislabeling. Current methods used for determination of species and adulteration are time consuming. Rapid Evaporative Ionisation Mass Spectrometry (REIMS) combined with multivariate statistical analysis (Principal Component Analysis and Linear Discriminate Analysis) is an emerging technique for near real time characterization of tissues with no requirement for sample preparation [1,2]. Samples are analysed by direct cutting of the surface of the sample using hand-held sampling devices powered by an electrosurgical RF-generator; a monopolar cutting electrode (the iKnife) or bipolar forceps. The resulting "smoke" or aerosol generated is transferred to the mass spectrometer via a Venturi air jet pump-based ion transfer apparatus mounted in the orthogonal position relative to the atmospheric interface of a quadrupole time of flight mass spectrometer. Although mass spectra acquired from food samples, including a range of different fish and meat species or from different cuts of meat, look similar, the profile of the lipid components has been shown to be useful for classification purposes using multivariate statistical methods. Using these spectra, training samples were used to classify the reference groups to build PCA/LDA models. The models were verified with cross-validation and independent test sets. We present data that demonstrates the potential capability of the REIMS technique to accurately discriminate meat muscle samples from different species and for detection of offal in processed meat products. Results are provided immediately using prototype "real-time recogniser" software.

P-47 Multiresidue Determination of Pesticides in a Fatty Matrix: an Alternative Cleanup for QuEChERS Extracts Prior to GC-MS and LC-MS Analysis

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In recent years, food safety laboratories have adopted new and simplified sample preparation methods designed to reduce analysis time and related costs, as well as to increase throughput. For example, the QuEChERS methods for fruits and vegetables require only minutes for sample preparation and replace prior methods that took hours or days. In this study, this type of simplified sample preparation is applied to pesticide analysis in avocado, a fruit matrix of very high lipid content. A typical avocado contains 10 - 15 % fat and about 1 % total phospholipids. In the QuEChERS extraction, significant amounts of the fat and phospholipids are co-extracted along with the target pesticides. The presence of these co-extracted substances can lead to chromatographic interference, contamination of the GC injector and column, contamination of the column and other components of the UPLC system and contamination of the mass spectrometer itself. To avoid these complications, a cleanup step is recommended prior to the instrumental analysis. This is typically performed using dispersive SPE with mixed sorbents, often with cumbersome multi-step centrifugation. In this study an Oasis PRiME HLB cartridge was used for a simple one-step pass-thru cleanup to effectively remove fats and phospholipids. Recovery data are presented for 37 pesticides registered for use on avocado in various world markets.

P-48 Novel Residue Analysis of Various Food Samples using GCxGC-HRMS with Encoded Frequent Pulsing™

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Environmental contaminants are a diverse group of compounds that come with many challenges when it comes to detecting and properly identifying each of the various compound groups. Add in the complications due to sample matrix interference, and confidently identifying environmental contaminants within a sample matrix can be a formidable task. By combining the separation power of comprehensive two-dimensional gas chromatography with a high resolution Folded Flight Path (FFP®) TOFMS that uses Encoded Frequent Pulsing (EFP™) technology, the ability to analyze samples

with a resolving power greater than 25,000 at full mass range, sub ppm mass accuracies, acquisition rates up to 200 spectra per second, and sub-picogram limits of detection is possible.
The addition of EFP technology with specialized real time decoding algorithms provides the ability to increase extraction frequency of the instrument to 20 kHz thus increasing the duty cycle without sacrificing spectral performance. To evaluate the performance of this new technology, a typical set of performance standards was first tested with and without EFP enabled to develop a baseline for sensitivity testing using prototype instrumentation. Then a complex mix of environmental standards was tested to evaluate the sensitivity of the instrument with regard to various compound types. This was then followed by the analysis of some pesticide residue matrix spiked samples to test 'real-world' performance using comprehensive two-dimensional gas chromatography coupled with a high resolution multi-reflecting TOFMS prototype with EFP. Select examples of tomato, strawberry, grape, or cabbage will be presented.

P-49 Glyphosate analysis in soy beans, corn and sunflower seeds by HPLC with post-column derivatization and fluorescence detection

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Glyphosate is a broad spectrum herbicide widely used around the world. Monitoring of Glyphosate in crops and water is mandated in many countries. We describe a sensitive and robust HPLC method for analysis of Glyphosate in soy beans, corn and sunflower seeds. This method utilizes a simplified sample preparation procedure that has proven to be effective even for challenging matrices.

P-50 Analysis of N-Methyl Carbamate Pesticides in Food by HPLC with Post-Column Derivatization and Fluorescence Detection

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Carbamate pesticides are widely used around the world to protect agricultural produce. In addition, they are used in homes, gardens and industrial applications. The main route of exposure for people to N-Methyl Carbamates is through food pathways, so pesticide use in food crops is strictly regulated.
As part of FDA's pesticide monitoring program, individual lots of domestic and imported foods and feeds are sampled and tested for pesticide residues in order to enforce the tolerances set by the EPA. Methyl carbamates are separated using a reversed-phase column and then react with o-Phthalaldehyde and a mercaptan after hydrolysis to form a highly fluorescent derivative. This post-column reaction is the basis for EPA Method 531.2 and AOAC official Method 985.23. The "QuEChERS" (Quick, Easy, Cheap, Effective, Rugged, and Safe) method is a single step sample extraction and salting out technique that is combined with dispersive SPE clean-up for multi-residue pesticide analysis. AOAC official Method 2007.01 utilizes QuEChERS extraction and clean-up for wide range of pesticides in food matrices. This method abstract demonstrates that dispersive SPE can be successfully used in combination with post-column derivatization and fluorescence detection for analysis of carbamates in food.

P-51 Extraction of Acrylamide from coffee and potato chips (crisps) using supported liquid extraction (SLE+) prior to LC-MS/MS Analysis

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Acrylamide analysis has increased in recent years due to its neurotoxic and carcinogenic properties. It is a small polar analyte with limited functionality leading to difficulties with extraction, purification and chromatography from various matrices. There are many methods for acrylamide measurement in the literature involving two separate solid phase extraction procedures which are expensive, complicated and time consuming. Approaches such as reversed phase and mixed-mode SPE have been used to selectively retain specific matrix components, or porous graphitic carbon as a more retentive phase to bind acrylamide. This poster demonstrates a novel simple, sensitive, cost effective and rugged method for the analysis of acrylamide in coffee and potato chips (crisps) using supported liquid extraction followed by LC-MS/MS analysis.
Coffee and potato chips were spiked with acrylamide at various concentrations. Matrix extraction was performed using widely accepted procedures: coffee beans were ground and extracted into hot water; potato chips were combined with water, rotated for an hour and centrifuged.
Supported liquid extraction was evaluated using modification of matrix pH prior to loading on to ISOLUTE SLE+ 200 µL capacity plates and 1 mL capacity columns. Extraction solvents were investigated based on varying polarity and solvent

properties: DCM, MTBE, EtOAc and combinations with tetrahydrofuran.
All extracts were analysed using a Waters ACQUITY UPLC system coupled to a Quattro PREMIER XE triple quadrupole mass spectrometer. Positive ions were acquired using ESI operated in positive MRM mode.

P-52 Evaluation of Methylisothiazolinone (MI) Extraction from Sunscreen using Supported Liquid Extraction prior to GC/MS Analysis.

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Methylisothiazolinone (MI) is an antimicrobial preservative that is used in a variety of personal care products, such as sunscreens, lotions, cosmetics etc. MI is a cytotoxin and there exist major concern over sensitization and allergic reactions as well as cell and nerve damage. A percentage of the population is at risk from contact dermatitis upon exposure to MI at sufficient concentrations. In July 2015, the European Commission adopted a ban of MI as an ingredient in leave-on cosmetics. The complex nature of the matrix in terms of various additives provided an interesting challenge, as MI is very water soluble but only partially soluble in organic solvents. In order to extract MI from the sunscreen, various combinations of organic/aqueous solutions were investigated. Optimization of the supported liquid extraction protocol involved investigation of loading volume and elution solvent combination. Various solvents such as DCM and MTBE provided analyte recoveries greater than 90%. However, visual extract cleanliness demonstrated some non-volatile material which resulted in incomplete evaporation, potentially due to the co-extraction of ethylene glycol additives. The optimal extraction solvent of 50/50 hexane/EtOAc demonstrated high analyte recoveries, greater than 90%, RSDs below 10%, good visual extract cleanliness and no interference on the SIM trace. Method performance was compared using vortex mixing to the use of a Biotage Bead Ruptor 24, which provided a far more homogenous extract. Calibration curves were constructed spiking MI into sunscreen from 50-750 ng/mL, where coefficients of determination (r^2) greater than 0.99 were observed.

P-53 Development of a Cleanup Method Incorporating a Novel SPE Media for the Analysis of Patulin in Apple Juice Using LC-MS/MS

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Patulin is a mycotoxin produced by Aspergillium and Penicillium fungal species commonly found on rotting apples. Although not a particularly potent toxin, patulin has been shown to be genotoxic and potentially carcinogenic, requiring regulation and analysis in apple-based products. Recommended maximum limits for patulin are set globally at 10 µg kg^{-1} in apple juice, 25 µg kg^{-1} in solid apple foods, and 10 µg kg^{-1} in apple-based infant food. Mycotoxins are a structurally diverse group of toxic metabolites produced by several strains of fungi found on food crops. Their potential to cause harm to humans, crops and farmed animals means that a wide range of food matrices need to be tested for mycotoxin contamination. The diversity of both analyte structure and food matrix produces a significant analytical challenge. Traditionally, mycotoxins have been analyzed using multiple methods, each one optimized for a single mycotoxin or group of very closely related toxins. The increasing adoption of liquid chromatography-tandem mass spectrometry (LC-MS/MS) based analysis facilitates a multi-analyte approach. Here we present work on the development of simple catch-and-release sample preparation method using a novel solid phase extraction (SPE) column for the determination of patulin contamination of apple-based products. The extraction method demonstrates high recovery of patulin whilst the accompanying HPLC method effectively resolves hydroxymethylfurfural, a significant endogenous interference. We are able to demonstrate patulin recovery of 101% and an LOQ of 10 µg kg^{-1} with an analytical working range of 2 to 200 µg kg^{-1} meeting regulatory requirements for patulin maximum residue limits.

P-54 Development of a Multiclass Cleanup Method Incorporating a Novel SPE Media for the Analysis of Mycotoxins in Grain Using LC-MS/MS

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Mycotoxins are a structurally diverse group of toxic metabolites produced by several strains of fungi found on food crops. Their potential to cause harm to humans, crops and farmed animals means that a wide range of food matrices need to be tested for mycotoxin contamination. The diversity of both analyte structure and food matrix produces a significant analytical challenge. Traditionally, mycotoxins have been analyzed using multiple methods, each one optimized for a single mycotoxin or group of very closely related toxins. The increasing adoption of liquid chromatography-tandem mass spectrometry (LC-MS/MS) based analysis facilitates a multi-analyte approach. Here we present work on the development of simple catch-and-release sample preparation method using a novel solid phase extraction (SPE) column for a variety of mycotoxin classes commonly encountered in grain providing cleanup for multiple mycotoxins: aflatoxins, ochratoxin A, fumonisin B1, T-2 toxin, HT-2 toxin, zearalenone, ergocornine and ergocryptine. The developed method was successfully applied to the analysis of mycotoxins in wheat, maize and barley. The method is capable of reducing matrix effects to levels enabling simultaneous measurement with a targeted LC-MS/MS method demonstrated by analyte signal/noise >10:1 at LOQ. The method demonstrates linear responses ($r^2 > 0.998$) over the working range. We demonstrate determination of 11 mycotoxins at or below regulatory requirements of current EU and US legislation with recoveries of between 71 and 110% at LOQ.

P-55 Quantitate Analysis of Leachable Contaminates of Bottled Water at Elevated Temperatures

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In a world where convenience is the norm and even sought after, packaged food and bottled drinks are ingrained in everyday life. Specifically, the consumption of bottled water is an ever growing trend as there is a growing concern about the safety and quality of drinking water. With the increased consumption of bottled water, questions arise – 'Is bottled water really better for you?' 'Is there a concern of contaminates leaching from the plastics?' The FDA has specific regulations in place for bottled beverages. These regulations are based on the EPA drinking water regulations for the quality of water used for bottled water. The International Bottled Water Association (IBWA) and the European Federation of Bottled Water (EFBW) set strict guidelines for bottled water manufacturers in the United States and globally. These strict guidelines are limited to production and storage. In *EFBW: Guide to Good Hygienic Practices for Packaged Water in Europe*, it states "specific temperature monitoring controls during transportation are generally not required" [1]. It also states that "outside storage is acceptable, if under cover, shrink-wrapped, or similar, and for limited periods only, less than 24 hours" [1]. Due to the concern of leachable contaminants in bottled water stored at elevated temperatures, studies have been conducted to quantify the leachable compounds from the plastic bottles. Usually the list of compounds is very specific. The intent of this study is to determine the amount of BPA and other common plasticizers leached from bottled water left in a car on summer day.

P-56 Accuracy Evaluation of a Free, Web-Based Tool for Gas Chromatographic Modeling for Multi-Class Chemical Contaminants Analysis

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Gas chromatography (GC) method development can be a tedious and resource-intensive process. Although MS and tandem-MS detection have reduced the need for baseline separation of all compounds, separation is still required for isobaric compounds, and optimized resolution is desirable. The trend toward analysis of large, multi-class analyte lists makes gas chromatographic method development more challenging.
This poster will introduce the Pro*EZ*GC® Chromatogram Modeler - a free, web-based application capable of producing model GC chromatograms under user-defined instrument conditions for hundreds of target compounds across several compound classes. This program generates and optimizes modeled chromatograms by employing a database of empirical data to predict elution temperature on a given stationary phase for each compound under varying capacity, flow, and temperature conditions. By using this tool, the time required to develop a new chromatographic method can be reduced from days to minutes, and the program can aid in existing method optimization through confirmation of predicted retention times and testing established conditions for additional analytes.
The focus compound classes for this presentation include pesticides and PCBs. An optimized solution generated by the modeler will be presented, and the predictive accuracy of the modeler will be thoroughly explored in terms of analyte retention time and resolution. Additional considerations for use of the modeler will also be presented including resolution constraint parameters, effects of injection technique on model accuracy, and use of mass spectral library data to aid in optimization through identification of mass spectral interferences.

P-57 Shoot-and-Dilute Gas Chromatography-Mass Spectrometry: Polycyclic Aromatic Hydrocarbons Quantification in Tea using Modified QuEChERS Extraction and No Sample Cleanup

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Food contamination with toxic polycyclic aromatic hydrocarbons (PAHs) occurs by exposure to environmental contamination and during food preparation, especially heat processing like smoking, grilling and roasting. While classic sample extraction methods such as Soxhlet and Pressurized Fluid Extraction (PFE) yield excellent recoveries for PAHs, they require expensive equipment/glassware, are solvent- and resource-intensive, and are relatively slow. The QuEChERS sample preparation method avoids these problems but produces a comparatively dirty sample. There are notorious problems associated with splitless injection of dirty samples, most notably for PAHs is decreasing response. This can occur very quickly with real samples, especially without exhaustive sample cleanup.

Methods explored here included streamlined sample preparation and Shoot-and-Dilute gas chromatography-mass spectrometry (GC-MS). A modified QuEChERS extraction was paired with a simple silica solid-phase extraction (SPE) cleanup. In addition, sample extracts without cleanup were also analyzed in order to determine if sample cleanup was necessary when combined with Shoot-and-Dilute GC-MS/MS. This work demonstrates that the ruggedness and sensitivity of Shoot-and-Dilute GC-MS/MS allows quantitation of the EU 15+1 PAHs in extracted tea samples without the need for sample cleanup. Incurred values at sub 10 ng/g levels were determined and proved similar to values determined via splitless injection GC-TOFMS. Ruggedness was demonstrated by less than 20% RSD of PAH relative response factors for over 100 injections of tea extracts with no cleanup. The combination of split injection and highly sensitive GC-MS/MS allowed samples with low PAH levels to be quantified without sample cleanup and, at the same time, prolonged system performance.

P-58 Prolonging GC-MS/MS Performance for Pesticide Analysis: Shoot-and-Dilute Injection and Analyte Protectants

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In gas chromatography–mass spectrometry (GC-MS), most problems occur on the front end, at the GC inlet, where compounds can degrade during hot splitless injection, active compounds can be irreversibly adsorbed to inlet liner surfaces, and nonvolatile material from dirty samples can compromise the transfer of less volatile compounds of interest from the inlet to the GC column. Two strategies to mitigate these issues were evaluated. One approach is to use split injection, “Shoot and Dilute”. Increased flow through the inlet during split injection minimizes residence time inside the inlet liner, which decreases compound degradation and adsorption, and maintains acceptable data quality longer. The second strategy to overcome GC inlet problems is to use “analyte protectants,” which are essentially volatile and chromatograph-able masking agents such as sugars, diols, etc., that are co-injected with each sample and standard to temporarily occupy active sites in the GC inlet liner and column.
Both strategies were tested with multi-class pesticides and compared against a typical splitless injection method without use of analyte protectants for QuEChERS samples. For Shoot and Dilute, viability of split injection based on detectability of a wide range of analytes was determined. Performance of analyte protectants and split injection were evaluated by peak shapes, initial relative response factors (RRFs) and associated RSDs. Ruggedness of both well-behaved and problem pesticides was evaluated by RRFs and RSDs for repeated matrix injections. It was determined that active compounds benefited from analyte protectants most for splitless injection while reactive compounds benefited most during split injection.

P-59 Improving the Analysis of Phenylurea Herbicides in Drinking Water and Soft Drinks using Automated Solid Phase Extraction as a Preparation for HPLC-UV Analysis

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The application of phenylurea herbicides is very important to increase yields of agriculture products, such as wheat, fruits and cotton. These compounds also have non-agricultural uses as well, such as preservatives in paints. However, the phenylurea herbicides, especially diuron, have been listed by U.S. Environmental Protection Agency as “known/likely” carcinogens. These herbicides are very water soluble and mobile in soils, which leads to contamination of surface water, ground water and eventually drinking water.
Accurate and consistent monitoring of these substances is important to public health. Screening for these compounds is vital to ensure water treatment systems are operating properly and risk of exposure is minimalized. Public water sources

are also used for the production of commercial products such as soft drinks. Since the same sources are used in these goods, screening for phenylurea herbicides is a priority to protect consumers.
The adoption of sample prep automation can increase the accuracy and reproducibility of results. Using equipment for solid phase extraction increases workflow efficiency and reduces the risk of errors causing costly retesting. While there is no established maximum residue limit (MRL) in drinking water, the phenylurea herbicides are on the US EPA CCL3 list which may result in official regulation. The typical MRL for foods is as low as 10 ppb in some areas of the world. This poster will demonstrate how automating the sample preparation process will help accurately and consistently identify and quantitate low levels of phenylurea herbicides.

P-60 Enhanced Food Safety Separations using Superficially Porous Particle Column Technology

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Superficially porous particles (SPP) have been proven to provide fast and efficient separations. These particles feature a solid, impermeable core enveloped by a thin, porous layer of silica that decreases the diffusion path and reduces peak dispersion. When combined with highly selective stationary phases, the result is significant improvements in efficiency, sensitivity, and run time over fully porous particles (FPP) of similar dimension. SPP columns can be substituted into existing methods to provide enhanced performance without changes in instrumentation or for the development of new assays. Several relevant food safety applications such as aflatoxins, pesticides, and veterinary drugs were developed with fast run times using SPP columns. For example, four aflatoxins can be eluted in less than 1.5 minutes using the Raptor™ ARC-18 with detection by liquid chromatography coupled with mass spectrometry (LC-MS/MS) in positive ion mode. In addition 204 pesticides were analyzed in 9.5 minutes by maximizing peak capacity over the entire gradient to ensure accurate detector response and quantitation. Finally, 61 veterinary antibiotic drugs were separated in 7 minutes on Raptor™ C18 by LC-MS/MS with polarity switching. Restek LC columns offer the speed of superficially porous particles with the resolution of the highly selective stationary phases, allowing peak separation and faster analysis times to be achieved without expensive UHPLC instrumentation.

P-61 Comparison of Sample Cleanup Methods (GPC and EMR-Lipid) for Multiresidue Pesticide Analysis in Avocado by GC/MS/MS

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QuEChERS is a sample preparation method widely used for pesticide residue analysis because of its simplicity and low solvent requirements. However, when the sample contains more than 3% fat, additional sample cleanup or an alternative method is required to remove this excess fat. One of the well-established techniques is Gel Permeation Chromatography (GPC) that removes the lipids from the extract by size exclusion chromatography. Although achieving good sample cleanup, this technique takes more time, requires additional steps (like solvent exchange) and uses more solvent. Enhanced Matrix Removal - Lipid (EMR-Lipid) is a new sorbent for removing lipids that can replace the traditional C18 dispersive SPE step. Avocado fruit was selected in this study because it contains about 20 % fat making it difficult to clean up with traditional QuEChERS and dispersive C18 SPE. Samples were fortified at 3 levels with 38 GC-amenable analytes from 12 different classes of pesticides. The extract, after the QuEChERS salting out step, was divided into two fractions with one part going through GPC and another to a 15 mL tube containing 1 g of EMR-Lipid. Recovery and reproducibility of both techniques were compared and results showed the possibility of having quicker sample cleanup, like QuEChERS, with fat removal as good as GPC.

P-62 Combined contaminant and multi-residue testing by LC/MS/MS in spices and herbal food supplements

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Routine screening and quantitation for pesticides but also other contaminants is important to increase the safety of food. During transport and storage these products are also affected by fungal infections raising the importance for testing for natural toxins. In this presentation we show a new rapid routine approach for combined screening and quantification of pesticides, mycotoxins, dyes and pyrrolizidine alkaloids in spices and herbal food supplements. Different spices and dried herbal food supplements were homogenized and extracted using QuEChERS with and without dispersive SPE clean-up. The extract was chromatographically separated on an Agilent 1290 Infinity Series UHPLC system combined with an

iFunnel triple quadrupole mass spectrometer operated with dynamic MRM and fast polarity switching. The final method included 267 pesticides, 11 mycotoxins, 28 pyrrolizidine alkaloids, and 6 dyes. For method performance characterization samples were spiked with all target compounds at relevant concentrations. Low levels of detection have been achieved by the optimized UHPLC method combined with a highly sensitive triple quadrupole LC/MS system. Lower limits of quantitation for most pesticides were below 10% of their maximum residue limit even with higher dilutions. With a 10 to 20-fold sample dilution, matrix effects were minimized and accuracies for most of the targeted compounds were within the desired range of 70 to 120%. Several pesticides, mycotoxins and pyrrolizidine alkaloids were identified in the spices and herbal food supplements in concentrations of 10 to 100 µg/kg. All results will be presented.

P-63 Analysis of Multiple Pesticide Residues in Fruits and Vegetables using GC/Q-TOF and EI Accurate Mass Pesticide Library

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The screening of a broad scope of pesticides in food requires untargeted data acquisition and good sensitivity to meet regulatory requirements on maximum residue levels (MRL). Accurate mass GC/Q-TOF serves as a fit-for-purpose tool for this demanding GC/MS application in pesticide analysis laboratories, as it offers untargeted acquisition of full scan EI mass spectra of all GC amenable pesticides at low concentrations. The rich accurate mass spectral information allows more comprehensive data analysis when unexpected new pesticides emerge. It is also desired to identify and quantify a large variety of pesticides within one run for improving efficiency. In this study, we used a novel GC/Q-TOF and accurate mass pesticide library based workflow for screening and calibrating 120 pesticides in peach and avocado samples. The GC/Q-TOF method with backflushing capability is retention time locked (RTL) to those library RTs. The screening of pesticides used automated software compound identification, with compound verification assisted by RT, ratio of fragment ion abundance and mass accuracy. At lowest spiking level of 5 ng/mL, we identified >110 those spiked pesticides in both food extracts, with instrument precision RSD ≤ 10% obtained for > 100 pesticides in avocado, the more complex matrix in this investigation. The screening workflow also dynamically selects quantifier of each identified pesticide to build quantification method. With an innovative algorithm for extended dynamic range, the majority of pesticides yielded linear calibration curve fitting coefficient (R^2) of ≥ 0.99 in the matrix matched calibration from 5 to 200 ng/mL.

P-64 Maintaining Sensitivity and Reproducibility with the Self Cleaning Ion Source for Pesticides in Food and Feed

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The global agricultural industry uses over a thousand pesticides for food and foodstuffs cultivation. Producers are compelled to use pesticides to meet the growing demand for reasonably priced food, resulting in the need for pesticide residue monitoring in commodities worldwide. Concurrently, simple sample preparation practices, such as QuEChERS are routinely used for the analysis of food and feed samples, often leaving significant amount of matrix in the exactas. Analytical laboratories are challenged by this matrix, which with time negatively affect the response of the analyzed pesticides, and eventually leads to source cleaning. Utilizing Agilent's Self Cleaning Ion Source (SCIS) reduces the need for manual source cleaning while allows for the analysis of complex samples without losing sensitivity and reproducibility. The Agilent Self Cleaning Ion Source utilizes carefully monitored hydrogen gas (H_2) introduction to the source, controlled by Agilent's MassHunter Data Acquisition Software. The appropriate H_2 flow (in the µL/min range) under proper environments generates conditions that clean the surfaces of the source, the detector, and other components. These actions aid in maintaining a stable detection milieu and provide for response stability of the pesticides in difficult matrices. Approximately 150 pesticides with various functional groups and vapor pressures were analyzed in two different and difficult matrices, honey and spinach. Matrix optimized MRM transitions for the pesticides were utilized for the analysis comparison of performance with and without the use of the SCIS.

P-65 Accurately Identify and Quantify A Hundred Pesticides in a Single GC Run

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The global agricultural industry uses over a thousand pesticides for the production of food and foodstuffs. More and more methods are being created to analyze the extensive list of target pesticides. Analytical laboratories are thus

strained to evaluate and quantitate hundreds of pesticides in a single run. Currently GC-MS/MS MRM analyses use time segments with predefined sets of MRM transitions for each segment. As sample complexity increases (i.e. quantifying low levels of hundreds of pesticide residues in a wide diversity of food matrices) the ability to utilize dynamic MRM (*d*MRM) provides laboratories with the capability to tackle large multi-analyte analysis and to accurately quantify trace quantities of pesticides from high-throughput methods. The analysis was conducted on an Agilent 7890B GC and 7010 Series Triple Quadrupole GC/MS. The Agilent 7010 MS/MS High-Efficiency EI source was run at 300 °C (quadrupoles at 150 °C), with a gain of 10, and an emission current of 100 µA. Agilent's MassHunter Data Acquisition was utilized for the development of the *d*MRM acquisition method. Approximately 150 various pesticides were analyzed in two different and difficult matrices, honey and spinach. Matrix optimized MRM transitions for the selected pesticides were utilized for the analysis. The MRM transitions were imported into the *d*MRM acquisition software and optimized to maintain a constant cycle time and a constant sampling rate across all peaks. By utilizing the *d*MRM method, hundreds of individual pesticides, their internal standards and qualifier ions, can be accurately identified and quantified in a fast GC run.

P-66 Significant improvement in GCMS screening of pesticides by use of a High-Efficiency Source and spectral deconvolution

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Routine pesticides residues analysis in environmental and food samples requires low-level detection with confident identification, which may be accomplished by the use of GCMS in full scan mode and spectral deconvolution with a program such as AMDIS. Improved full scan screening capability is made possible by the use of a GCMS system equipped with a High-Efficiency Source (HES), which increases the number of ions that are created and transferred into the quadrupole analyzer. This increase in response translates into better sensitivity and thus more targets found with good NIST library matches. Pesticide identification in food at 10 ng/g is now possible for many residues using scan mode as was demonstrated by analysis of tomato extract spiked with over 200 pesticides. Analysis was performed using the Agilent 5977B GC/MSD equipped with an HES operated in scan mode. The HES was then exchanged with a standard extractor source and the analysis repeated. Spectral deconvolution was by Deconvolution Reporting Software (DRS) using AMDIS with a custom library. NIST hits (reverse match) were determined as well. At the low concentration of 10 ng/g, or 10 pg injected, 38 AMDIS targets were identified using the HES as compared to zero using the standard extractor source. At the 100 ng/g level, almost twice as many residues were identified; 164 in the case of the HES compared with 91 when using the extractor source. These preliminary results demonstrate that positive identification in full-scan mode for many targets in food at a concentration of 10 ng/g is possible.

P-67 Aflatoxin Analysis in Infant Formula with Enhanced Matrix Removal — Lipid by LC/MS/MS

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Mycotoxins are secondary metabolites of fungi and are considered to be one of the most prevalent contaminants in food and feed supplies globally. Aflatoxin M1 is the primary aflatoxin found in milk and is produced when cows ingest and metabolize feed contaminated with aflatoxin B1. The Food and Drug Administration (FDA) has established an action level of 0.5 µg/kg for aflatoxin M1 in milk while the European Commission has set a maximum level of 0.025 µg/kg in infant formula. A sample preparation method has been developed for five aflatoxins (M1, G2, G1, B2, and B1) in infant formula using a QuEChERS extraction followed by cleanup with Enhanced Matrix Removal – Lipid (EMR—Lipid) dispersive solid phase extraction (dSPE) and LC/MS/MS analysis. This method delivers excellent recoveries (88–113%) and precision (RSDs = 1.3–13.6%) for all aflatoxins at three concentration levels. Due to the extensive matrix removal, limits of quantitation (LOQs) for this method were extended below regulatory limits for both the U.S. and Europe. EMR – Lipid is quick, easy, and can be effectively applied to multiclass mycotoxin analysis in high-fat samples. This simple and robust method requires minimal equipment and expertise, which promotes easy implementation in food laboratories.

P-68 Determination of Mycotoxins in Peanut with Enhanced Matrix Removal – Lipid and Liquid Chromatography-Tandem Mass Spectrometry

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Mycotoxins are secondary metabolites produced by various species of mold which grow on many agricultural

commodities, either in the field or during storage. These mycotoxins are regulated in China and other countries worldwide. A method for the quantitative determination of 12 mycotoxins in peanut has been developed. A QuEChERS extraction is followed by cleanup with Enhanced Matrix Removal – Lipid (EMR – Lipid) dispersive solid phase extraction (dSPE). Samples were analyzed with a 1290 Infinity LC System coupled with a 6460 Triple Quadrupole Mass Spectrometer. The dynamic calibration ranges for these compounds are 0.5 to 500 ng/mL. Overall recoveries are between 70 to 120% with RSD values below 15% for three concentration levels. Limits of quantification (LOQs) for this method are below current mycotoxin regulations in China. This method demonstrates that QuEChERS and EMR – Lipid can be used in combination for a quick and effective extraction of mycotoxins in high-fat samples such as peanut.

P-69 Targeted Veterinary Drugs Screening in Animal Tissues Using Agilent EMR QuEChERS Kit and 6470 Triple Quadrupole Mass Spectrometer

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The analysis of veterinary drug residues in animal tissues is an important aspect in food safety. However, it presents analytical challenge due to the large number of trace-level analytes to be identified in the complex matrices. Sample preparation and sensitive LC-MS/MS method are both critical for the successful screen of veterinary drugs in animal tissues. EMR QuEChERS kit is specifically designed to remove fatty materials in the matrix, and is thus optimal for animal tissue sample preparation. Newly developed 6470 triple quadrupole LC-mass spectrometer, with its improved Q1 ion transmission optics and detector, is suited to meet the challenge. In our experiments, about 90 veterinary drugs were simultaneously detected using a sub two micron C18 column and the mobile phases consisting of water-acetonitrile with formic acid and ammonium fluoride as modifiers. Sensitive detection was attributed to the efficient matrix fat removal and the chromatographic separation of analytes from matrix in a 17-minute gradient. At least two MRM transitions were monitored for each analyte. Calibration curves were generated for all analytes spiked from 0.1-100 ng/g in four matrices: pork, salmon, beef and beef liver. All calibration curves show good linearity with most of R^2 >0.99. At LLOQ level, at least 4 out of 6 replicates had to meet the accuracy requirement between 80 and 120%. Precision was evaluated at LLOQ level with 6 replicate injections, and most of them showed single digit %RSD.

P-70 An Optimal Method for the Analysis of Pesticides in a Variety of Matrices

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The global agricultural industry uses over a thousand pesticides for the production of food and foodstuffs. Producers require pesticides to meet the increasing demand for reasonably priced food both in and out of season. This growing demand has increased the use of pesticides and expanded poor agricultural practices elevating risks in the food supply and environment. Analytical laboratories are then strained to evaluate and quantitate hundreds of pesticides in a wide range of matrices. Not only are laboratories faced with time constraints, but they also face matrix interferences that interfere with their ability to accurately identify and quantitate the multitude of pesticides. Utilizing matrix optimized MRM transitions for these pesticides provide laboratories with optimal acquisition methods for their pesticides analysis. Approximately 150 various pesticides were analyzed in several different matrices that embrace leading global commodities. Five varieties of matrices were examined; these included a clean commodity (yellow onion), a high sugar commodity (honey), a high starch commodity (rice), a high pigment commodity (spinach), and a high oil/fat commodity (olive oil). Each matrix was extracted with its specific QuEChERS methodology. Calibration standards were prepared at concentration levels ranging from 0.5 pg/µL to 500 pg/µL. All analyzed pesticides obtained a %RSD ≤ 20%, required by the SANCO guidelines. With the use of the 7010 Series Triple Quadrupole GC/MS in MRM mode, the analysis was able to confirm pesticide residues at the low ppb level even in the most complex extracts (i.e. olive oil, spinach, and rice).

P-71 Two-gram Incurred Food Samples Using Mini-QuEChERS, Cryomilling and GC/MS/MS Analysis with a High Efficiency Ion Source

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A novel approach in food analysis involves mini-QuEChERS extraction method with cryomilling and only 0.5 uL injection volume into the GC/MSMS. Incurred fruits and vegetables underwent homogenization and cryomilling, which reduced particle size and provided a uniform 2 g sample, 87% less commodity than the 15 g sample required by AOAC QuEChERS protocol. The mini-QuEChERS protocol for 2 g sample with cryomilling was evaluated for pesticide residue analysis

relative to the 15 g sample required in the validated method. The extraction efficiency in several matrices showed that the results from the original 15 gram sample size are comparable to the 2 gram mini-QuEChERS and the smaller sample size can be implemented without additional changes in equipment and or protocol. For example, results in cucumber were as follows: chlorothalonil, 51.5.0 ng/g (15g) versus 44.4 ng/g (2 g); metalaxyl, 37.0 ng/g (15 g) versus 35.0 ng/g (2 g). The mini-QuEChERS sample preparation procedure is easier to handle, uses far less solvent, salts and labeled standards and was shown to produce comparable results. Additional labeled standards for problematic compounds like captan and folpet is an acceptable option with the mini-QuEChERS since it is not cost prohibitive as it would be for a 15 gram sample size.
Cost savings ranged between 42-48% due to less solvent, sorbent and labeled ISTD. Use of a 0.5 µL injection and high efficiency ion source reduced cost-per-sample through less frequent maintenance and allowed for improved limits of quantitation for incurred residues in a variety of fruits and vegetables.

P-72 Improving Pesticides Analysis on GC/MS/MS for Complicated Samples by Increasing Matrix Removal

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GC/MS/MS analysis has been widely applied in routine pesticides analysis, however, co-extracted matrix from complex samples, especially lipids, often hinder data quality. Sample matrix could accumulate in the GC flow path, generating active sites that interact with target analytes, especially for labile compounds, causing irreproducible results and challenging the reliability of the analysis. The QuEChERS sample preparation technique has been widely employed for preparing samples prior to GC/MS/MS analysis. Traditional and new dSPE cleanup materials were compared for the removal of lipids and other co-extractives, resulting in significant differences in matrix removal efficiency and selectivity. When applied in a QuEChERS workflow, a new dSPE cleanup material provides dramatically higher matrix removal efficiency than traditional dSPE cleanup methods, while delivering excellent analyte recovery and reproducibility. Improvement in sample matrix removal allows for better method sensitivity and selectivity due to less interferences and better signal noise ratios. Additionally, cleaner samples reduce matrix accumulation on the GC flow path, improving instrument reproducibility and GC column and consumables' lifetime. Lastly, high matrix removal allows large volume injections (LVI) on the GC/MS/MS system while maintaining acceptable chromatographic integrity and analyte reproducibility. This will certainly provide more flexibility on GC/MS/MS method development in order to achieve the desired method sensitivity. By increasing matrix removal through appropriate sample cleanup, the analysis of pesticides in complex, fatty sample types using GC/MS/MS is more reliable, and data will demonstrate the performance advantages of cleaner samples for selected pesticides in avocado using GC/MS/MS.

P-73 Complete Workflows for Food Pesticide Residue Laboratories Using Tandem and High Resolution Accurate Mass (HRAM) LC/MS Instrumentation

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Food pesticide residue laboratories face significant analytical challenges. Growing target compound lists, large numbers of samples, wide varieties of matrices, and decreasing limits of detection are pressuring labs to become more efficient than ever before. In addition, customers often require more information on contaminants that are not on any target lists that may be a threat. Clearly, an integrated solution using both HRAM and tandem MS is needed to address this ever-changing landscape.
Routine quantitative workflows using representative matrices are presented on both LC tandem and HRAM instrumentation. Method validation for the triple quadrupole method according to the EU guidelines will be discussed for a robust, high-throughput targeted analysis as a 'ready-to-go' solution for labs. In addition, HRAM analysis with data-dependent acquisition will be presented. In this mode, full scan accurate mass information is stored for each sample, and the isolated precursor/product ion pairs serve as a means of confirmation. The use of accurate mass compound databases along with web-enabled searches via easy-to-use processing software allow the user to look beyond target lists and screen for other contaminants.
Performance parameters of a complete routine tandem MS method were tested, and demonstrated that a vast majority of the measured compounds can be analyzed with a high degree of confidence. Limits of quantitation less than 10ppb (ug/kg) were achieved for greater than 97% of compounds in all matrices tested. Method performance was tested via analysis of FAPAS CRM samples. The HRAM results showed similar limits of quantitation and excellent discrimination against matrices at a mass resolution setting of 70,000. The MS/MS spectra acquired in data-dependent mode could be used for confirmation, and the stored full scan data could be processed retrospectively for screening analysis.

P-74 Meeting the European Commission Performance Criteria for the Use of Triple Quadrupole GC-MS/MS as a confirmatory Method for PCDD/Fs in Food and Feed Samples

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Legislation in the European Union previously required the confirmation and quantification of PCDD/Fs in contaminated samples by gas chromatography/high resolution mass spectrometry (GC-HRMS) instruments, considered the "gold standard" approach. However, recent technological advances in gas chromatography/triple-quadrupole mass spectrometry (GC-MS/MS) technology have allowed high sensitivity and selectivity to be achieved. These improvements have led to GC-MS/MS being considered a reliable tool that can be used to control the maximum levels for PCDD/Fs in food and feed as a full confirmatory method

P-75 An assessment of GC Orbitrap MS technology for the routine screening and quantification of pesticide residues in food.

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The analysis of pesticide residues in food is challenging because of the high number (typically >800) of substances that need to be analysed in a diverse range of complex matrices, at low cost and with a fast reporting time. In order to achieve this within a routine environment, sensitive and selective LC and GC triple quadrupole MS systems are traditionally used. However, the operating mode of choice when targeting such large numbers of compounds using triple single or quadrupole mass spectrometers is either selected ion monitoring (SIM) or selected reaction monitoring (SRM). Both SIM and SRM have limitations, especially in terms of the number of pesticides that can be analysed in one injection. In addition, only the pesticides added in the SIM/SRM method will be targeted, without the possibility to detect additional contaminants. To overcome these limitations it is important to employ a mass spectrometer system able to analyze food samples in full scan with similar performance as triple quadrupole techniques. A generic acquisition based on full scan MS is more straightforward and provides additional information compared with multiple reaction monitoring by triple quadrupole mass spectrometry. It also increases the scope of the analysis, as target compounds are selected post acquisition. In order to obtain sufficient selectivity in the full scan mode, high resolution/high mass accuracy MS instruments are required.
In this study we evaluated the recently introduced GC-EI-Orbitrap MS system for the routine screening of pesticides from different chemical classes in a variety of fruits and vegetables. The criteria used for a positive detection (retention time, mass accuracy, isotopic pattern, fragment ions, spectral matching) were evaluated. An assessment of the identification points was made at different concentrations and resolving powers. The quantitative performance (linearity and residuals) was assessed following the screening. Initial results show that the system and software is capable of meeting the required criteria at very low limits of detection, well below the maximum residue levels.

P-76 The analysis of polar ionic pesticides by ion-exchange chromatography tandem mass spectrometry: the possible solution to a longstanding problematic analysis?

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Polar ionic pesticides, such as glyphosate, perchlorate, chlorate and the like, often occur as residues in food, but are not always included in pesticide monitoring programs, simply because they are not 'amenable' to generic multi-residue methods. The introduction of the Quick Polar Pesticides (QuPPe) Method by the European Reference Laboratory for single residue methods (EURL-SRM) has enabled more laboratories to conduct analysis for at least some of the polar pesticides. Still, the absence of a liquid partitioning step, or 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 challenging. Analysts attempt to mitigate these issues by analyzing a single extract a number of times, using different chromatographic columns and conditions. These separation conditions are often less than ideal and the large amounts of co-extractives often contaminate the low capacity columns to cause variation in retention time and a decrease in the ruggedness of the method.

The application of high resolution ion-exchange chromatography with high capacity columns, coupled to a triple quadrupole mass spectrometer can overcome the issues experienced with other chromatographic techniques. Using the IC-MS/MS approach for direct analysis of QuPPe extracts, low limits of quantification (typically < 5 ng/g) , and associated repeatability (typically < 20%) have been achieved for chlorate, perchlorate, glufosinate, N-acetyl glufosinate, 3-MPPA, glyphosate, AMPA, Fosetyl-Al, phosphonic acid, ethephon, HEPA and more, in a single analysis. Further details on separation, quantification and validation in various matrices will be presented.

P-77 Determination of Pesticide Residues in Drinking Water Using Automated Solid Phase Extraction with GC-NPD

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Organophosphorous pesticides have been commonly used in agriculture to protect crop yield and in public health applications to prevent the spread of disease from mosquitos (e.g. West Nile Virus). Organophosphorous compounds have relatively fast rate decomposition when compared to organochlorine compounds and may be preferred for use due to their lower probability of persistence in the environment. However, use of organophosphorous compounds has also become a concern as large doses of these compounds have been demonstrated to cause neurotoxicity. These compounds can enter the body through inhalation, ingestion, or skin contact and inhibit cholinesterase, the enzyme responsible for breaking down the neurotransmitter acetylcholine. When cholinesterase is inactive, acetylcholine builds up in neurons causing overstimulation of the nervous system which may result in muscle weakness, convulsions, and respiratory failure, Due to the these effects and that fact that organophosphorous compound residues may be present in drinking water sources, regulatory agencies such as the U.S. EPA have established methods for monitoring their levels to ensure public as well as ecological safety.
This study evaluated the detection of trace amounts of organophosphorous pesticides in water and optimizes the conditions for solid phase extraction and chromatographic analysis using gas chromatography with nitrogen phosphorous detection. Eight different organophosphorous compounds were extracted and detected in finished drinking water with a sensitivity of 0.02 -0.1 ug/L with extraction recoveries ranging from 83 – 100%. The methods presented in this study achieve a rapid, simple extraction with small quantities of solvent used and minimal operator involvement.

P-78 Increasing Extraction Efficiency of Pesticides & Dioxins from Wet Samples using a Novel New Polymer during Accelerated Solvent Extraction

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Accelerated solvent extraction (ASE) is a high-temperature and high-pressure extraction technique that is widely used for sample extractions in the environmental, chemical and food analysis industries. Extraction efficiencies when extracting volatile or semi volatile analytes from wet solids are often low, as the analyte of interest may partition between the extracting solvent and the water phase. Traditional pre or post extraction methods of heat evaporation cannot be used for volatile and semi-volatile compounds. Drying techniques that involve mixing the wet samples with an inorganic salt that has a high affinity for the aqueous phase are unsuitable for in-cell extractions. Drying methods with inorganic salts suffer from the limitations of clumping or precipitation making post extraction clean-up difficult. Off-line drying methods like freeze drying are extremely tedious and time consuming.
This study presents the use of a novel new polymer designed to remove moisture from wet samples like soil, tissue and food products and increase the extraction efficiency of volatile and semi volatile compounds. The polymer has a high capacity for water removal and does not suffer from some of the limitations of clumping or precipitation observed in some of the traditional drying methods. Data showing recoveries of organochlorine pesticides, polychlorinated dibenzo-p-dioxins, polychlorinated dibenzofurans, and polychlorinated biphenyls in different matrices will be presented.

P-79 China as a Global Partner in Generation of Pesticide Residues Dataset

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Dow AgroSciences (DAS) has recently developed a global package for registration of a new active on rice. The global data package for rice included residue data generated in the United States, Australia, Brazil, Argentina, Japan and China. The goal of this global data package was to harmonize the treatment rates (Good agricultural practices, GAP), the analytical methodology and the resulting maximum residue levels (MRLs) in rice. Analytical methods for residue analysis were maintained across geographies allowing for flexibility for each laboratory's resources. The synchronization of MRLs is important to insure no trade barriers exist between exporting and importing geographies. Another important aspect

of this regulatory approach is the utilization of China as a partner in the generation of OECD Good Laboratory Practice (GLP) - compliant data for registration support. As a globally emerging economy, China represents a large and important market for rice. The training and cooperation of DAS and Chinese colleagues will be discussed, as well as the advantages gained by utilization of the local Chinese data in the global data package.

P-80 Bridging extraction efficiency information for data generation residue methods and QuEChERS enforcement methods using samples with in-grown residues—a case study.

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To comply with the SANCO/825/00 rev. 8.1 guideline requirement, "*the extraction procedures used in residue analytical methods for the determination of residues in plants, plant products, and foodstuff (of plant and animal origin) should be verified for all matrix groups for which residues ≥ LOQ are expected*". Using the development phase of a new Dow AgroSciences pesticide as a case study, the poster will describe the analytical laboratory's strategy to ensure that extraction procedures of various residue analytical methods are adequate to extract all relevant residues identified in the carbon-14 fate and metabolism studies while maintaining reasonable sample analysis throughput. The technologies and opportunities to streamline the process of generating extraction efficiency information for both pre-registration data generation methods and post-approval enforcement methods will also be discussed. The advantage of using LC-MS/MS over conventional radioactive detection technique will be demonstrated. In addition, the poster will also discuss options, in terms of extraction efficiency assessment, to ensure guideline compliance when new matrices are to be included in the methods when the pesticide use is expanded. Furthermore, the suitability of the multiresidue QuEChERS methods for accurate tolerance/MRL enforcement using representative matrices with in-grown residues will also be evaluated.

P-81 2α-Hydroxyursolic acid inhibits cell migration and invasion in MDA-MB-231 human breast cancer cells via EGFR-dependent PI3K/Akt signaling pathway

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Breast cancer is the most common cancer diagnosed among women in the United States. Epidemiological studies suggest that increased consumption of fruits and vegetables has linked to reduced risk of developing breast cancer. Previous work from our lab demonstrated 2α-hydroxyursolic acid, one of the major triterpenoids isolated from apple peels, had potent anti-proliferative activity against human breast cancer cells. However, the mechanisms of anti-migrated and anti-invasive activities are not completely understood. The objective of this study was to investigate the mechanisms of action of 2α-hydroxyursolic acid in inhibiting cell migration and invasion in MDA-MB-231 human breast cancer cells. 2α-Hydroxyursolic acid was isolated and purified from apple peels as reported previously in our laboratory. The results showed that 2α-hydroxyursolic acid significantly inhibited EGF-induced MDA-MB-231 cell migration and invasion in a dose-dependent manner at the concentrations without cytotoxicity. The activities of MMP-2 and MMP-9, critical enzymes for cancer cell migration and invasion, were dramatically inhibited in a dose-dependent manner. Western blot analysis indicated that 2α-hydroxyursolic acid significantly inhibited EGF-induced phosphorylation of EGF receptor (EGFR) and Akt. Furthermore, 2α-Hydroxyursolic acid suppressed EGF-mediated nuclear protein levels of NF-κB, c-Jun and c-Fos, known as transcriptional factors, leading to down-regulation of VEGF expression. 2α-Hydroxyursolic acid has been shown to have potent activity in inhibiting migration and invasion in MDA-MB-231 human breast cancer cells via EGFR-dependent PI3K/Akt signaling. These results are important in understanding anticancer activity of fruits and vegetables and potential application of 2α-hydroxyursolic acid in the prevention of breast cancer.

P-82 Washington State Department of Agriculture Chemical and Hop Laboratory workflow to ensure high quality data in single and multi-residue methods

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The WSDA Chemical and Hop Laboratory analytical testing serves a wide range of clientele: USDA's Pesticide Data Program, various WSDA programs, other state agencies, and the public. While implementing a quality assurance unit (QAU) can be tedious work, a well-established QAU ensures all data is of high quality and produced in a timely manner to meet the needs of our clients. Currently we employ both single and multi-residue screens for trace and formulation analysis of pesticides and other chemical residues in various matrices (fruits, vegetables, soil, water, foliage, feed, fertilizers, hops, cannabis, clothing, swabs, etc.). Two essentials of our QAU include proficiency test samples (internal and external) and continual monitoring (via statistical process control charts) of analytical instrument performance,

surrogates, lab fortified blanks, and internal standards; both of which help identify any trends within our processes. We also regularly apply LEAN principles to our laboratory workflow to improve the overall efficiency of our operation (including methods) without sacrificing quality. As an ISO 17025 accredited laboratory, our QAU is an important part of our everyday tasks to ensure our clients are provided with reliable and reproducible data.

P-83 Composting: A Biological Process for Aflatoxin Decontamination in Agricultural Environment

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In developing countries, there is a high occurrence of aflatoxin contamination in peanuts due to climate conditions and handling practices. Contaminated peanut wastes and shells are often used as soil amendments and mulching materials, which re-introduces aflatoxins and aflatoxin-producing molds into subsequent farming seasons.
This research evaluated whether composting can be used to control aflatoxin contamination in agricultural environment by using peanut meal with a high level of aflatoxin contamination as a model matrix.
The peanut meal was uniformly mixed with deionized water. The samples were inoculated with either one of the 3 commercial starters alone or in combination with a commercial accelerator. The control was peanut meal without the starters or accelerator. Samples were incubated at 40°C in a water bath for 6 W. Compost temperature, pH and ammonia concentration were documented twice a day during the process. Aflatoxin B1, B2, G1 and G2 were quantified at the end of each week using high performance liquid chromatography. *Aspergillus flavus* counts were enumerated on malt extract agar incubated at 25°C for 3-7 d.
The composting resulted in a significant reduction in the amount of B1, B2, G1 and G2 in peanut meal during the 6 W. The amounts of B1, B2, G1 and G2 reduced from 275.03 to 53.59 ppb, 30.47 to 6.98 ppb, 11.41 to 0 ppb, and 3.14 to 0 ppb, respectively. *A. flavus* counts reduced from an average of 103 CFU/g to the undetectable level. Compost temperature, pH and ammonia content ranged from 22.1°C to 60.6°C, 4.41 to 7.94, and 0 to ≥ 500 ppm, respectively at different stages of the composting process.
The research demonstrates that composting is effective means to decontaminate aflatoxin and control *A. flavus* growth in peanut wastes. The process has the potential to reduce the level of aflatoxin contamination in agricultural environment.

P-84 Efficacy of soil amendments in reducing leaching of Agriculture Chemicals to soil and ground water in sub-tropical region of Uttarakhand, India - An Ecofriendly Approach

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A lab and field experiment was conducted to examine the effect of different soil amendments in preventing the leaching of commonly applied pesticides (Chloropyrifos, Cypermethrin, Imidacloprid and Carbendazim) in vegetables and cereal crops. Fresh yard manure (FYM), Gypsum, Cereal straw (CS), Press manure compost (PMC) @ 5 t ha^{-1} and Fresh cow dung (FCD) @ 0.5 t ha^{-1} were applied on surface soil (0 to 15 cm) in columns along with pesticides @ 2 ppm level concentration. In order to observe the leaching of pesticides in field, piezometer were installed and the surface soil was examined for pesticide concentration at different time intervals i.e 10 days before and 30 and 60 days after application of pesticides and amendments in soil. Monitoring of water table depth, electric conductivity (EC) and nitrate content in ground water was also done along with the pesticide residue analysis.
Quantitative analysis was done using GC and HPLC. The results indicated that leaching of agrochemicals like chloropyrifos, cypermethrin, carbendazin and imidacloprid were reduced by 75 to 80% by application of amendments in comparison to control. FCD served as the best amendment in preventing leaching of pesticides in water probably due to the high organic carbon content which increases the binding capacity of the nonpolar agrochemicals. The pH and EC of water were not affected by amendment application and the nitrate content in leached water was also well within the permissible limits. The approach is overall ecofriendly and economical in preventing ground water pollution.

P-85 Development of a FTIR Method to Identify Herbicides and their Low Volatile Counterparts

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2-4 Dichlorophenoxyacetic acid (2-4D) and dicamba are commonly used herbicides in agriculture. Soybean plants are resistant to the effects of 2-4D but not dicamba. Cotton plants are resistant to dicamba but are susceptible to the effects of 2-4D. Drift is a well-known issue with herbicide application. Companies have recently released low volatile versions of dicamba and 2-4D in hopes to reduce the amount of drift that occurs. Regulations have been recently required farmers to switch to low volatile herbicides but an analytical method to differentiate between the herbicides and their low volatile form is needed in order to enforce these regulations. Cotton with no herbicide applied, cotton with 2-4D applied, and cotton plant with LV 2-4D added were also analyzed using FT-IR. Separation between the all three sample types was acquired. The first three principle components combined explains 99% of the variance in the data set for the cotton samples. Soybean with no herbicide applied, soybean with dicamba applied, and soybean plant with LV dicamba applied were analyzed using FT-IR. Good separating for all three sample types was achieved. The total percentage of the first three principal components was 100%. The 2-4D and dicamba as well as their low volatile counterparts were all distinguishable using the FT-IR.

P-86 Applying lipidomics for elucidating biomarkers and the role of environmental stressors leading to pansteatitis outbreak in fish across South Africa

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Lipids are important in numerous biological functions including as cell membrane structural components, as signaling molecules, as energy storage molecules, in immune function, and as targets of oxidation. Environmental stressors causing oxidative damage such as trace metals, can alter the lipid profile observed. This is the first study applying lipidomics to wildlife populations to determine biomarkers and etiology of environmental stress induced disease. A lipidomics workflow consisting of lipid extraction, reverse phase chromatography, and high resolution mass spectrometry, was used to determine biomarkers and potential causes of the pansteatitis (yellow fat disease) outbreak causing an alarming number of crocodile deaths coinciding with massive fish die-offs in national parks across South Africa.
Linking changes in lipid profiles to sources and etiology of disease using lipidomics may shed light on the diverse variables leading to pansteatitis in South Africa, such as mining operations releasing a suite of trace metals. Trace metals and lipid profiles were measured in hundreds of Mozambique tilapia (*Oreochromis mossambicus*) and tens of African sharptooth catfish (*Clarias gariepinus*) across various isolated aquatic environments (Loskop, Flag Boshielo, and Kruger National Park). Depending on the species and gender different lipids were up or down-regulated in diseased populations indicating lipid profile changes based on behavior or effects specific to gender and species. Disease versus healthy tissues (tilapia swim bladder and adipose) separated in PCA, showing drastic differences between healthy and diseased tissue lipid content.

P-87 The determination of plasticizers and other chemical pollutants, from industrial point sources in Wallingford and North Haven with an emphasis on the pollutant's impact to indigenous fish populations in the Quinnipiac River

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The Quinnipiac River presents itself as a delicate ecosystem supporting various wildlife species. Pollutants, specifically plasticizers and phthalates to these waters can be harmful to the ecosystem and its native species. Our lab has previously collected water samples from a drainpipe located off Toelles Road in Wallingford, CT that was analyzed by Gas chromatography-mass spectroscopy (GC-MS). Several compounds were identified, most notably phenothiazine (PTZ), hexamethyl melamine, and methyl vinyl ether. Plasticizers are endocrine disrupting chemicals (EDCs). These EDCs can interrupt normal endocrine function, leading to a physiological imbalance normally, with negative side effects for the organism. In order to better understand the biological impact of these compounds, Fundulus heteroclitus (mummichog) was used. Mummichogs are a native fish to the Long Island sound and can be found up into the Quinnipiac River making it an ideal organism to determine the biological impact of these compounds. The fish were allowed to acclimate to the laboratory tanks for one week and then treated with differing, environmentally relevant concentrations of PTZ, 0.5 ppm, 1.0 ppm, and 2 ppm, for seven days. GC-MS was performed before and after each treatment to show relative concentrations as well as the degradation of the compound over the seven days. Following PTZ treatment, the brain, liver and gonads were dissected of from each treatment tank, samples were pooled and RNA was extracted via RNeasy MIDI kit (Qiagen) and converted to cDNA using iScript cDNA synthesis kit (Biorad). Quantitative PCR (qPCR) was then

utilized to measure gene expression for hormone receptors and samples were normalized to β -actin. Preliminary analysis of the hormones receptor genes Glucocorticoid, Androgen, Progesterone, Estrogen α, Estrogen β A, and Estrogen β B receptor revealed that glucocorticoid receptor and Estrogen β B receptor mRNA levels increased following treatment.

P-88 Use of a Multianalyte Method by Undergraduate Biology and Chemistry Students for the Analysis of Phthalate Esters and Industrial Contaminants in River Water

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The Quinnipiac River is a regional water resource which arises in central Connecticut and flows southward into Long Island Sound. The river is commonly used by the public for fishing, despite the presence of organic pollutants from several manufacturing companies that have permits to discharge their chemical waste into the river. Due to these industrial discharges, the river is an excellent model for teaching undergraduate students how to sample and analyze environmental pollutants while collecting data for public safety.
The multianalyte method we chose for the analysis of river water samples uses solid phase micro-extraction followed by detection with gas chromatography with a mass selective detector for compound identification. The method is capable of testing for the presence of phthalate esters and other volatile compounds (MW 60-275) in water. Example target analytes which can be detected are benzyl butyl phthalate, bis-2-ethyl hexyl phthalate (diethyl hexyl phthalate), dibutyl phthalate, diethyl phthalate, and other volatile industrial compounds such as phenothiazine, an additive in polymer production.
Results, from May to November 2015, showed the presence of low levels of phthalate esters, specifically dibutyl phthalate, benzyl butyl phthalate, diethyl hexyl phthalate, at very low levels, less than 50 parts-per-billion in river water. In May 2015, phenothiazine was found in samplings, entering the river from a discharge pipe, at levels in the 0.5-3 ppm range. In June and July 2015, samples from the same pipe showed the presence of what appeared to be monomers, or fragments of monomers, from a methyl vinyl ether based polymer.

P-89 Identification of Surfactants in Hydraulic Fracturing Fluids by Ion Mobility Mass Spectrometry

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Non-ionic surfactants are used in hydraulic fracturing, which is a new technology for extracting oil and gas from impervious shale. Shale is fractured with 10-20 million liters of water per well, which contain non-ionic surfactants as lubricating agents and friction reducers. Half of the water returns to the surface for deep well disposal and the remainder is left behind in the geologic formation. Because of possible groundwater contamination, it is important to identify and "fingerprint" the organic compounds with new analytical tools, such as ion mobility mass spectrometry and accurate mass. The Agilent Model 6560 ion mobility mass spectrometer was used for these studies equipped with a 1290 UHPLC system using positive and negative ion electrospray with accurate mass at less than 2 ppm and resolving power of 25,000 at m/z 922.0098. Three groups of non-ionic surfactants, polyethylene glycols, polypropylene glycols, and polyethylene glycol carboxylates, were identified and databases developed that combined accurate masses with ion mobility drift times for the identification of the three families of surfactants. Drift times were converted to collisional cross sections by the software as an identification feature for each of the surfactants, which can be used along with their liquid chromatographic retention time and their accurate masses for the identification of the surfactants . Furthermore, heatmaps were generated that coupled with drift times and collisional cross section data for the surfactants were used to easily distinguish different wells from the DJ basin.

P-90 Analysis of Organic Marker Compounds and Hazardous Organic Compounds by GC/MS to Identify Contamination, Counterfeiting and Adulteration of Spices

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Food adulteration and counterfeiting continues to grow as a worldwide issue of food safety and economic concern. Spices are one of most commonly adulterated and counterfeited agricultural products in the US. Our previous study determined there were extensive elemental and heavy metals contamination and adulteration in spices. Many of our spice products were identified possibly being highly adulterated or contaminated by metals. In our follow-up Organic study, we focused on the organic markers and toxic organic compounds in our common spices and botanicals (Black

Pepper, Red Pepper, Cinnamon, Mustard Seed, Cumin, and Turmeric) in various forms (i.e. spices, teas, condiments and supplements) to determine if these products appeared to be adulterated from an organic compound standpoint as well as an elemental standpoint.
Cryogenic grinding and microwave extraction were employed in sample processing. Samples were extracted for the primary and secondary marker compounds native to each spice group and for any potentially toxic organic compounds (dyes, preservatives, pesticides & industrial residual chemicals). The concentration and identity of compounds were compared across the groupings and to cited concentration references for each marker or compound. Low concentrations of critical markers were found in low cost spice and botanical samples indicating potential adulteration. Samples that were previously suspect by ICP-MS examination were confirmed to be adulterated or economically compromised by reduced or absent concentration of these critical primary and secondary marker compounds. High levels of potentially toxic chemicals were also found in some of the previously suspect spice and spice product samples.

P-91 An Automated Extraction Solution for the Determination of Melamine and Cyanuric acid in Milk Based Products

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The determination of Melamine and Cyanuric acid go hand in hand with one another. Melamine is an industrial chemical used in the manufacturing of amino resins and plastics. Cyanuric acids are produced by the hydrolysis of raw or waste melamine, followed by crystallization. Melamine is used as a profitable adulterant in milk based products to increase the protein content and thus increase the commercial value of the adulterated substance. In recent years Melamine and Cyanuric acid has been found in infant formula and other milk based products. The presence of Melamine and Cyanuric acid is highly regulated around the world.
In this study, the performance and versatility of the AutoMate-Q40, work station platform, was evaluated for the extraction of Melamine and Cyanuric acid in milk based products. A Liquid Chromatography (LC) coupled to a Triple-Quadrupole Mass Spectrometer (LC-MS/MS) was employed for the detection of these adulterants. Quantification was based on external calibration curves. By using the AutoMate-Q40 to streamline this extraction, it provides us with appropriate analytical results, falling in the established method guidelines (recovery range of 70-120% and a %RSD <20%) for the target compounds.

P-92 Time and Cost Effective Methods for Reducing Background Noise and Signal Suppression in Problem Matrices for Residue Analysis by LC-MS/MS

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Several clean-up methods are compared for background reduction, analyte recovery, and cost effectiveness in order to successfully analyze a wide variety of multiclass multiresidues in difficult matrices including: Citrus, Dried Habanero Peppers and Tobacco. An ISO accredited QuEChERS method, as well as a dilute and shoot approach are analyzed in conjunction with different filtration techniques for residue analysis by LC-MS/MS for minimal number of steps, speed, reduced reagent use and reduced cost.

P-93 Quantitation of Terpenes in Cannabis Products Using LC-MS/MS and Atmospheric Pressure Chemical Ionization

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With the recent legalization of cannabis in several states, there is a growing need for robust, cost-effective analytical methods to facilitate routine testing. While testing of potency (and for pesticide and herbicide residues) is important, manufacturers of cannabis products also need fit-to-purpose analytical methods that provide information on the sensory profile of their products to ensure lot-to-lot consistency. Here, we present an LC-MS/MS method that uses atmospheric pressure chemical ionization (APCI) for the analysis of terpenes (major determinant of aroma) in cannabis products. Examples of accurate quantitation are shown for a variety of cannabis products.
At least 200 terpenes have been identified in Cannabis sativa (cannabis), with unique strains presenting different terpene profiles. The terpenes present have a well-defined role in the perceived aroma and user preference for specific cannabis strains. Moving beyond a role in sensory perception, recent studies suggest that many cannabis terpenes have pharmacological properties of their own and may also act synergistically with cannabinoids. As such, sensitive, selective, accurate and economical analytical methods are needed to assess these key compounds.
Historically, terpenes have been analyzed by GC-MS due to their predominantly aliphatic composition. However, labs performing routine testing of cannabis will need to test potency and, once regulations are established, test for pesticides

and herbicides. LC-MS/MS represents an ideal analytical platform to address all of these testing needs. Herein, we present an LC-MS/MS method that uses APCI and the budget-friendly SCIEX Triple Quad™ 3500 LC-MS/MS system for the analysis of terpenes in cannabis products.

P-94 Advantages of Non-Targeted Information Dependent Data Acquisition using LC-HR-MS/MS followed by Targeted Data Processing to Screen for Pesticides in Cannabis Samples

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Like any agricultural product, cannabis has the potential to be attacked by pests or pathogens resulting in treatment with insecticides, acaricides, fungicides, and potentially other crop protection agents. Many of these types of contamination are known in other crops, where tolerance levels are established through health risk analysis.
Pesticide use in cannabis production is of concern for several reasons. While residues on the marketed product are important metrics for quality, it may be difficult to associate trace residues with human health effects, or these correlations may take years of careful medical research to become detected. Perhaps more importantly, the creation of rational guidelines for pesticide use can serve to protect workers in the production system and the environment. As a high value crop, cannabis will no doubt prompt some growers to use any and all measures to maximize yields, regardless of burdens or risks placed on employees, customers, or their surroundings. This should be prevented by appropriate registrations, inspection, and residue analysis. As such, sensitive, selective, accurate and economical analytical methods are needed to screen cannabis products for pesticide residues.
Using LC-MSMS analysis provides the sensitivity and selectivity for these low levels using (Multiple Reaction Monitoring (MRM). However, targeted MRM screening methods will detect only those compounds that are in the acquisition method, whereas a non-targeted Information Dependent Acquisition (IDA) method will detect any unknowns in the sample, and then the data can be processed with or without a target list.

P-95 Using LC-HR-MS/MS and Intuitive and Automated Software Workflows to Quickly Identify Unknown Compounds in Food Samples

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LC-MS/MS is a powerful analytical tool for the analysis of a wide molecular weight range of polar, semi-volatile and thermally labile compounds. Triple quadrupole-based mass analyzers are popular for targeted quantitation of hundreds of food contaminants in a single analysis because of their extra degree of selectivity and sensitivity when operated in Multiple Reaction Monitoring (MRM) mode.
Recent advancements in LC-MS/MS technology, including hybrid systems like quadrupole-quadrupole Time-of-Flight (QTOF), now provide the ability to perform targeted and non-targeted screening in food samples on a routine basis. Here we present results using a new method to identify unexpected chemical residues and contaminants in food using the SCIEX X500R QTOF system. Samples were extracted using a generic extraction method. LC separation was achieved using a generic reversed phase column and gradient of water/methanol and ammonium formate buffer. MS detection was performed using information dependent acquisition to simultaneously collect accurate mass MS and MS/MS information.
Unknown compounds were automatically identified by using a non-target peak finding algorithm followed by sample-control-comparison to separate matrix and sample specific signals from true contaminations. TOF-MS and MS/MS data for ions of interest were automatically processed using formula finding and searched against mass spectral libraries and online databases, such as ChemSpider, for identification. The SCIEX OS software offers an easy to use and intuitive workflow to tentatively identify unexpected chemicals in food.

P-96 Simultaneous Identification and Quantitation of Pesticide Residues in Food Samples using LC-HR-MS/MS

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Pesticides are widely used in agriculture to protect crops and to improve efficiency of production. Pesticide residues may pose a potential threat to human health. Modern analytical techniques, such as LC-MS/MS allow the screening for hundreds pesticide residues in food samples quickly, efficiently, and with excellent sensitivity and selectivity to meet global food trade guidelines and regulations.
Recent advancements in LC-MS/MS technology, including hybrid systems like quadrupole-quadrupole Time-of-Flight

(QTOF), now provide the ability to perform targeted and non-targeted screening in food samples on a routine basis. Here we present results using a new method to identify and quantify pesticide residues in food using the SCIEX X500R QTOF system. Samples were extracted using a QuEChERS method (Quick, Easy, Cheap, Effective, Rugged and Safe) and extract were diluted 10x to minimize possible matrix effects. LC separation was achieved using a polar modified reversed phase column and a mobile phase consisting of water/methanol and ammonium formate buffer. MS detection was performed using information dependent acquisition to simultaneously collect accurate mass MS and MS/MS information.
Limits of quantitation of 10 µg/kg were achieved for every compound after 10x dilution of the extract. Target compounds were automatically identified by matching retention time, accurate mass and isotope pattern of the molecular ion and MS/MS library searching using SCIEX OS software. In the same data processing step, compounds were quantified and unknown samples were flagged when a user-defined reporting level was exceeded.

P-97 Identification, Quantitation and Confirmation of Pesticides in Food Samples using LC-MS/MS and Ultra-fast Polarity Switching

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Recent regulations on food analysis require the screening for pesticides using confirmatory techniques, such as GC-MS(/MS) and LC-MS/MS. With more than 1000 pesticides of more than 100 compound classes there is a demand for powerful and rapid analytical methods, which can detect very low concentrations in food matrices. Here we present a high sensitivity and high selectivity LC-MS/MS method that combines Multiple Reaction Monitoring (MRM) quantitation with MRM ratios as a first step of identification. Samples were re-analyzed using LC-MS/MS using information dependent acquisition (IDA) of QTRAP® MS/MS spectra. MS/MS spectra were searched against extensive mass spectral libraries for high confidence confirmation.
Food samples, including a variety of fruits and vegetables were extracted using a QuEChERS procedure and injected into LC-MS/MS after extensive dilution to minimize or possibly eliminate matrix effects. LC separation was performed using a Phenomenex core-shell Kinetex Biphenyl column and a gradient of water and methanol and ammonium formate buffer with a total run time of 15 min. Detection was performed on the SCIEX QTRAP® 6500+ system using Electrospray Ionization (ESI). First injection was performed using the Scheduled MRM™ pro algorithm and ultra-fast polarity switching (5 msec) to reproducibly and accurately monitor approximately 600 transitions for the quantitation and identification of 300 pesticides.
In a second injection already identified pesticides were confirmed based on MRM-IDA-MS/MS analysis. The acquisition of full scan MS/MS spectra helped to reduce false positive findings. Data processing was performed using MultiQuant and MasterView software.

P-98 A Highly Selective and Sensitive LC-MS/MS Method for the Quantification of Gluten Proteins

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Gluten is a multi-protein complex located in the endosperm portion of wheat, rye, and barley grains that are commonly found in Western diets and are steadily becoming more prevalent in Eastern diets. Gluten ingestion has been linked to a number of gastrointestinal disorders, including celiac disease, wheat allergies, and non-celiac gluten sensitivity, with the epidemiologically relevant prevalence of these disorders estimated to be around 5% of the global population.
In 2013, the FDA established, among other criteria, a gluten limit of less than 20 parts-per-million (ppm) for foods that carry a gluten-free label (e.g., gluten-free, no gluten, free of gluten, without gluten).
Herein, we have developed and verified a selective and sensitive LC-MS/MS-based method for detecting and quantifying gluten signature peptides in a variety of food matrices. This method relies on the use of three MRM transitions for each unique gluten signature peptide released from the glutenin subunit (DY10). To increase assay precision, a stable isotope-labeled gluten standard was added to food homogenates with unknown gluten levels prior to enzymatic protein digestion. Assay performance was evaluated using raw cereal grains, as well as baked, dehydrated, and fermented products.
LC-MS/MS detects multiple, unique peptide markers simultaneously in a single injection, providing information on gluten content, as well as the identity of other grains (rye, barley, oats). Using this LC-MS/MS method, an accurate gluten concentration as low as 5 ppm can be obtained, with excellent repeatability (%CV) of less than 20%, along with information on a food product's grain composition.

P-99 Quantitation of Glyphosate and Other Polar Pesticides in Beer Samples using LC-MS/MS

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Glyphosate is a common broad-spectrum systemic herbicide used widely to kill weeds especially annual broadleaf weeds and grasses known to compete with crops. Usually Glyphosate, as it is very polar, undergoes FMOC derivatization by reacting the native glyphosate with fluorenylmethyloxycarbonyl chloride (FMOC-Cl) before analysis. This derivatization step complicates the analysis and there is a growing need for a method which can detect not only Glyphosate (and it´s major metabolite AMPA) but also Glufosinate and similar highly polar compounds, in their underivatized states.
Glyphosate is a topic with an extraordinary degree of public attention and concerns since the International Agency for Research on Cancer (IARC), a branch of the World Health Oragnization, classified glyphosate as a probable human carcinogen. Traces of glyphosate have been found in many foods (i.e. bread, beer) and also in human urine.
Here we present different LC-MS/MS methods to detect underivatized glyphosate and other polar pesticides. MS/MS detection was performed using a SCIEX QTRAP® 6500+ system operated in MRM mode in negative polarity ESI. Different published LC methods were evaluated and compared with respect to sensitivity, selectivity and routine use.
The method was successfully applied to quantify Glyphosate in beer after dilution and direct injection and also to detect polar pesticides in QuPPe extracts of soy and corn.

P-100 Identification of Artificial Colors and Dyes in Food Samples using LC-HR-MS/MS

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Artificial colors and dyes are used in food to make it visually more appealing and “flavorful” since people associate certain colors with certain flavors. However, some dyes are banned because they are toxic and carcinogenic. Analytical methods used to test for the presence of illegal colors and dyes in food include LC-UV and LC-MS/MS.
Here we present a method using the SCIEX X500R QTOF system to identify artificial colors and dyes in food samples. MS detection was performed using information dependent acquisition to simultaneously collect accurate mass MS and MS/MS information. Compounds were automatically identified by using a non-target peak finding algorithm followed by sample-control-comparison and statistical data processing to separate matrix and sample specific signals from true contaminations. TOF-MS and MS/MS data for ions of interest were automatically processed using formula finding and searched against mass spectral libraries and online databases, such as ChemSpider, for identification. The SCIEX OS software offers an easy to use and intuitive workflow to tentatively identify unknown chemicals in food.

P-101 Profiling of Hop-Derived Bitter Compounds in Beer using LC-HR-MS/MS and Statistical Data Processing

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Besides its sedative activity, beer has been attracting consumers over centuries due to its refreshing character, attractive aroma, and typical bitter taste. Aroma-active bitter compounds of beers have been thoroughly investigated in recent years, and it is agreed that the typical beer bitterness is caused by adding cones, pellets, or extracts of hop during the wort boiling.
During the wort-boiling process, a number of isomerization processes have been identified to be of major importance for bitter taste development in the final beer product.
Various approaches were taken in the past to measure bitter compounds in hops and beer to characterize the flavor of beer and to quantitatively determine “bitter units”.
Here we present a method to study the profile of hop-derived bitter compounds using LC coupled to high resolution mass spectrometry. Diluted beer samples were injected directly into the SCIEX X500R QTOF system. Data were processed using targeted lists of accurate masses of molecular ions but also in non-targeted fashion by performing statistical data processing (Principal Components Analysis, PCA). PCA returned accurate mass and retention time information which can be used to identify unknown flavor components.

P-102 Qualitative Analysis of 12 Food Allergens in a Single LC-MS/MS Injection

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A food allergy is an immune-mediated, adverse reaction to an antigenic protein. Even limited exposure to an antigen can provoke a significant reaction in sensitive individuals, causing rashes, itching and swelling in the mouth, nausea, vomiting, and asthma. Additionally, food allergies are the leading cause of anaphylaxis, an acute, potentially deadly allergic reaction. The prevalence and severity of food allergies are rising, with approximately 150 million people suffering from food allergies worldwide. Presently, there is no cure for food allergies, and sufferers must rely on the correct labeling of foods to avoid consuming allergens. Hence, the development of sensitive and accurate analytical methods to screen for the presence of allergens in food products is necessary for the prevention of potentially life-threatening health problems for allergy sufferers.
Herein, we present an LC-MS/MS method that detects and screens 12 separate food allergens simultaneously in a single injection. The allergens detected in this method were selected from the guidelines presented in the Codex Alimentarius, a resource developed by the United Nations' Food and Agriculture Organization (FAO) and the World Health Organization (WHO) to harmonize international food standards, including eggs, milk, peanuts, soy beans, and tree nuts (almonds, Brazil nuts, cashew nuts, hazelnuts, pecans, pine nuts, pistachios, and walnuts). Presently, this method can detect characteristic tryptic peptides from five of the major classes of allergenic foods at a detection limit of 10 ppm in a variety of food matrices.

P-103 Determination of Vitamin A and Vitamin E in Infant Formula

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Infant formula contains nutritionally beneficial levels of vitamins A and E. These vitamins play an important role in infant health and development. Therefore, monitoring vitamin A and E levels in infant formula is a crucial part of the quality control program for infant formula manufacturers.
The analysis of vitamins A and E in infant formula is challenging due to the nature of the matrix, the hydrophobicity of the compounds, and the number of vitamin A and E isomers present in a sample. In particular, resolution of the early eluting vitamin A palmitate isomers requires optimal HPLC conditions and high performance HPLC media. The AOAC official method 2012.10 outlines a normal-phase HPLC procedure utilizing both ultraviolet and fluorescence detection. Prior to HPLC analysis the reconstituted formula undergoes liquid-liquid extraction (LLE). This poster demonstrates the AOAC official method for the analysis of vitamin A and E in infant formula using optimized mobile phase conditions on a Luna® NH_2 HPLC column. The method provides adequate separation of all vitamin A and E isomers with an analysis time of less than 12 minutes.

P-104 Acrylamide from Coffee using Simplified Liquid Extraction (SLE) by HPLC

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Supported Liquid Extraction (or Simplified Liquid Extraction) is very popular in the clinical research industry however the technique is gaining popularity in other industries as a faster, easier, and more reliable alternative to liquid-liquid extraction. This study will investigate an application with implications for the food safety industry using Novum SLE to clean up and extract acrylamide from both instant and brewed coffee.

P-105 Rapid and Simple Extraction and Analysis of Vitamin D2 and D3 from Dietary Supplements Using QuEChERS and HPLC-UV

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Many individuals are looking to supplements for additional vitamins, minerals or nutrients that are not available in their daily diet. The need for label claim accuracy is both a quality and a safety concern, as these supplements are not regulated by the FDA. The use of Chromatography via HPLC-UV analysis is a great tool to accurately identify and quantitate the amounts of specific nutrients to ensure that what is consumed is what is reported on the label. Vitamin D specifically is a fat-soluble vitamin that has been known for its ability to enhance intestinal absorption of calcium, iron, magnesium, phosphate, and zinc. As part of the Quality Assurance (QA) process, dietary supplement manufacturers must quantify the components on the label claim. Presented here is a rapid and simple extraction procedure using QuEChERS salts and HPLC analysis using a Kinetex 2.6 µm Biphenyl Core-shell HPLC column.

P-106 Separation Solutions for Triglycerides in Food Fat and Oil by High Temperature GC Analysis

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Triglycerides are esters of glycerol with three fatty acids, and are naturally occurring in food. These compounds have relatively high molecular weights that increase with the degree of unsaturation, and are typically analyzed using low polarity GC stationary phases. Because the boiling point of these compounds are very low, high oven temperatures are essential to elute the analyte out of the stationary phase. Traditional columns used for this testing are limited to maximum oven ramp temperatures of approximately 370 °C. This could cause problems such as carryover of un-eluted high boiling compound in the following injection, excess degradation of the external column coating, and increased phase bleed, which leads to both reduced sensitivity and increased cost due to consumable replacement.
The present work focuses on the high temperature analysis of triglycerides in butter, olive oil, peanut oil and canola oil using GC oven temperatures as high as 400 °C. Methods for high boiling compounds utilized optimized ramp procedures and a thin film, low-polarity Zebron™ ZB-5HT Inferno™ GC column, which provided stability to 430 °C for the high oven ramp programs used. Considering the high boiling point of the triglycerides from different food sources, the method was optimized individually using thin film high temperature Zebron ZB-5HT Inferno columns. The study revealed preferred materials and methods that avoided external film degradation, prevented carryover, and improved sensitivity.

P-107 Determination of Melamine from Pet Food using Liquid-Liquid Extraction and GC/MS Analysis

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Melamine is a base that is classified as harmful if swallowed, inhaled or absorbed through the skin. Chronic exposure may cause cancer, reproductive damage, eye, skin and respiratory irritant and or renal dysfunction. Considering the ailments, it is very important to screen food for melamine before consumption. Food matrix is very complex and needs special attention in terms of sample clean up and even challenging are the basic analytes. So, proper extraction, derivatization and inert GC column selection are vital to get reproducible results and symmetric peak shape. We chose dog food as the matrix and spiked them with 100 µg/g melamine and related impurities. This was followed by extraction using Water Acetonitrile and diethyl amine. The extract was further derivatized using silylating agent. The clean sample was then analyzed by GC/MS using a specially deactivated Zebron ZB- 5MSPLUS GC column, which provided excellent peak shape and separation of melamine and related compounds. The derivatization process, optimized GC MS method and right column choice helped in achieving sharp and symmetric peaks which facilitated quantitation of melamine and related compounds from real food matrix.

P-108 Extraction of Cannabinoids from Brownies using QuEChERS Extraction and GC/MS Analysis

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Food matrix is very complex and needs special attention in terms of sample clean up. In the present study, we employed QuEChERS technique to remove matrix interferences from brownies. The resulting clean sample was then analyzed using a specially deactivated Zebron ZB-5MSPLUS GC column. This column provided excellent peak shape for Cannabidiol, Δ-9-Tetrahydrocannabinol and Cannabinol. GC MS analysis was performed by SIM mode to quantitate the cannabinoids. Quechers extraction not only helped in increasing the partition of the analytes in the organic phase but also helped removal of matrix interferences. Optimized GC MS method and right column choice helped to get sharp and symmetric peaks which facilitated quantition of the three analytes of interest from complex matrix like brownies.

P-109 A Rapid Screening Method for Analysis of Multi-Class Antibiotics from sausage using QuEChERS and LC/MS/MS

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Antibiotics consist of many different classes of compounds such as sulfa drugs, penicillins, tetracyclines, and cephalosporins, etc. These agents are used to treat infectious diseases for well over 70 years in humans. This usage has also been applied to food animals to control the bacterial harmful effect. In addition to this therapeutic use in food animals, antibiotics have been proven to promote growth when administered in small daily doses. The mechanism of this

phenomenon is unclear, but the use of antibiotics for growth promotion is on the rise and not well-publicized. According to US FDA, over 13 million kilograms of antibiotics approved for use in food animals were sold in the US and distributed to other countries in 2009. Over time, the daily use of low-dose antibiotics as feed supplements will promote antibiotic resistant bacteria. Furthermore, the subsequent consumption of the meat from these food animals can create the same phenomenon in humans and hamper the treatment of drug-resistant bacteria by conventional antibiotics. This improper use of antibiotics in food animals is an enormous concern to public health and safety. In this study, we present a rapid and sensitive multi-class screening method for the detection of multiple classes of antibiotics in sausage meat samples at maximum residue limit levels defined by Commission Regulation (EU) No 37/2010. Samples were prepared using a simple, yet effective extraction and cleanup procedure using Quechers. Extracts were analyzed using high efficient core-shell technology Kinetex® 2.6 µm XB-C18 HPLC column. Excellent signal-to-noise ratios were obtained at low spike concentration of 50 ppb and based on a small volume, 10 µm sample injection. This method was proven to be powerful for the detection of antibiotics in meat produced from food animals.

P-110 Analysis of Multiple Mycotoxins in Soybeans and Soymeal by LC-TOF: A Comparison of SPE and Modified QuEChERS Sample Preparation Methods.

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Mycotoxins in grains, feed and milk are regulated by almost every country in the world to assure the safety of food stocks for both humans and livestock. Mycotoxins have traditionally been analyzed by immunoassay techniques which are fast and simple but can suffer from poor sensitivity from matrix affects and are nonspecific when analyzing multiple aflatoxins. As regulations trend towards more selectivity and higher sensitivity many laboratories are moving to LC-MS techniques for analysis of mycotoxins in food. Although LC-MS can is very selective and sensitive, good sample preparation is required to clean-up and concentrate mycotoxins from complex food matrices prior to LC-MS analysis. In this study a PerkinElmer UHPLC coupled to the AxION 2 TOF MS was used for the simultaneous analysis and quantitation of multiple classes of mycotoxins extracted from soybeans and soymeal. A comparison of modified QuEChERS and SPE techniques was conducted by Analitus Laboratories to evaluate the best technique for sample preparation in these matrixes.

P-111 Gas chromatography coupled to triple-quad ICP-MS-MS for compound-independent quantification of organophosphate pesticides in honey bee products after miniaturized QuEChERS extraction

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The use of an elemental detector as powerful as inductively coupled plasma mass spectrometry (ICP-MS) have impacted several areas of research by increasing detection power and decreasing interferences. The powerful ionization source allows compound independent quantification of most metals and metalloids at sub-ppb levels. Although some biologically and environmentally relevant elements like S, P and Se suffer from intense isobaric interferences from atmospheric and plasma related sources, making traditional ICP-MS measurements not feasible. In recent years a technological breakthrough on elemental analysis was introduced with a triple quad configuration ICP-MS, which dramatically increase the capabilities of this analytical technique to quantify difficult elements by removing the interferences in a very controlled chemical environment. We use this state of the art new instrumentation to target organophosphate pesticides in honey bee products with very good results. For this, a gas chromatographer was directly coupled to an Agilent 8800-triple-quad ICP-MS-MS to monitor the hetero atom phosphorous as internal elemental tag in organophosphate pesticides. A miniaturized QuEChERS extraction and clean up procedure was optimized for bee wax, propolis, comb and pollen for GC-ICP-MS-MS analysis, and the results show recoveries of 75-105% for eighteen commonly used pesticides. The specific detection of phosphorous improved the quality of the obtained chromatograms for some of the most challenging samples, with limits of detection of 10 to 50 ng g^{-1} and most importantly compound independent calibration was obtained for fifteen out of the eighteen compounds of interest. Real samples from different geographic origins were analyzed, and coumaphos was detected in three of them. This new approach can reduce the amount of toxic waste generated by avoiding the stock of large amount of standards and its health hazard risks associated.

P-113 Do we still need to prepare matrix-matched calibration standards for GC-MS analysis of pesticide residues?

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The matrix-induced enhanced response effect in gas chromatography has been a problem for analytical chemists when susceptible organophosphorus pesticides were introduced at least 50 years ago. Matrix effects occur when relatively polar analytes (*e.g.* acids, esters, alcohols, ketones, amines, amides) interact with active sites in the inlet, column, and ion source in GC-MS. In solvent-only calibration standards, the analytes fill the active sites, causing their loss and a lower signal. However, when matrix components (or high concentrations of other susceptible pesticides) are also present in the analyzed solutions, other chemicals fill the active sites, leading to higher signal of the analytes and a high bias in quantification by solvent-only calibration standards. For official pesticide registration and regulatory enforcement methods, the EPA did not permit the use of matrix-matched calibration to overcome this problem until recently. However, the degree of matrix effects varies among matrices and within matrices, which introduces inaccuracies in the matrix-matching approach, independent of EPA policy. The method of standard additions has always been permitted and can lead to better accuracy, but this is terribly inconvenient. Matrix-matching is also very inconvenient since it requires matrix blanks of many types of samples, extra sample preparation, as well as extra costs in time, effort, supplies, and instrument maintenance needs. Analyte protectants can be added to all standards and extracts to maximize responses of susceptible analytes by filling the active sites in the system, but a good analyte protectant has yet to be found that compensates for matrix effects of late-eluting pyrethroids. In this study, we evaluated a combination of techniques, including analyte protectants, in the attempt to finally resolve this long-standing problem in pesticide residue analysis. Split injection with a deactivated liner in fast, low-pressure GC-MS/MS is also used to reduce the residence time of the analytes, thereby reducing interactions with active sites. For pyrethroids, pyridaben-d13 is employed as an internal standard to compensate for the similarly susceptible late-eluting pesticides. Additionally, automated mini-cartridge SPE cleanup is used to provide cleaner final extracts that undergo less extensive matrix effects than dispersive-SPE of QuEChERS extracts. Initial results using the overall approach look very promising, and in this poster, we shall present the validation results with and without matrix-matching for 72 representative analytes spiked at 10, 100, and 1,000 ng/g in 4 representative commodities (pear, orange, cilantro, and fish).

P-115 Reduction in Pipette Tip Consumable Cost and Waste Through Innovation

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As the number of samples processed in labs using high-throughput processes has increased, there has been a corresponding rise in the level of plastic consumables waste, such as pipette tips. The TipNovus high-throughput automated pipette tip cleaning system from Grenova enables laboratories to safely reuse sanitized tips and thereby reduce cost and waste output. The ability of TipNovus to sanitize tips was tested by comparison and validation studies of new and washed tips. All experiments were conducted using a Microlab STAR system with unfiltered CO-RE tips (Hamilton Robotics, Reno, Nev.). A TipNovus system washed up to four racks of 96 unfiltered CO-RE tips every 8-10 minutes, and the duration depended on the program. Red blood cell transfer for new tips and washed tips was evaluated by GC/FID. Chromatography from the sample transfer using the new tips was compared to that of the validation run using the washed tips. A correlation was obtained via linear regression. The model explained most of the variability in the data with an R2 value of 0.9872, indicating a strong linear relationship between results from the new and washed tips. Plasma samples transferred via the new and washed tips were run on a LC/MS/MS system. An R2 value of 0.9962 for this data demonstrated a strong linear relationship between results from the new and washed tips. Gravimetric volume verification from the transfer of 100 µL of distilled water produced a coefficient of variation below 1.0% for 20 consecutive wash cycles of the same tip.

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