52ND ANNUAL

NACRW

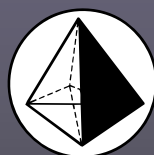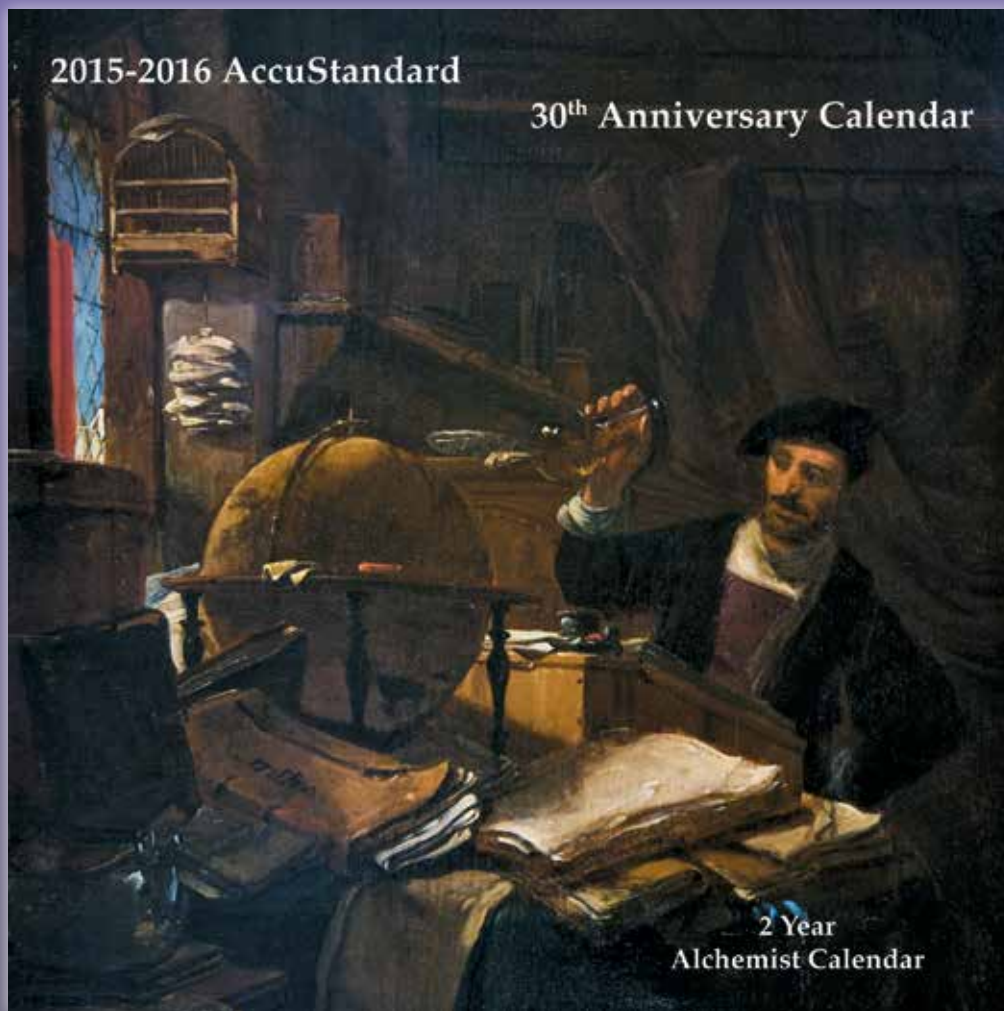NORTH AMERICAN CHEMICAL RESIDUE WORKSHOP

TradeWinds Island Grand Resort
St. Pete Beach, Florida
July 19-22, 2015

"Bringing Scientists Together to Develop and Validate Better Methodologies"

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FUTURE MEETING DATES

2016   July 17-20
TradeWinds St. Pete Beach

2017   July 23-26
Naples Grande Resort

2018   July 22-25
Naples Grande Resort

2019   July 21-24
Naples Grande Resort

July 19, 2015

Welcome Attendees, Exhibitors and Sponsors,

Welcome to the 2015 NACRW workshop. I hope you are ready for great science and a lot of fun!

Over the past year, I have had the honor of working with an outstanding and enthusiastic Organizing Committee and it is my pleasure to highlight some of the activities we have planned for you. We have worked hard to create and deliver the best technical program possible starting with the short course. The short course, Pesticide and Veterinary Drug Residues: Method Validation and Routine Quality Control, was requested by attendees last year and we are happy to have secured such great instructors in Ana Lozano and Eric Verdon. There is no doubt this will be a valuable course for their students.

The technical sessions span a variety of chemical residue related subjects as well as some special interest areas. As the mainstay of the workshop, many aspects of both pesticide residue and veterinary drug residue testing will be discussed. In addition, sessions on special topics like aquatic toxins, food fraud and adulteration and emerging chemical contaminants are represented. Back by popular demand are the Updates from Federal and State Regulatory Laboratories and the Mass Spectrometry Forum.

In addition to the oral sessions, do not miss the posters sessions and vendor seminars. The poster presentations are of equally high technical quality as the lectures. Poster authors will be presenting their posters at designated times and I am sure they are eager to discuss their work. This is a great opportunity to learn from and offer advice to your NACRW friends. We are pleased to offer vendor seminars that allow you to learn from experts about the latest and greatest developments as well as communicate your analytical needs.

When I first attended the workshop, I was amazed at the friendliness and comradery. We hope to continue this by offering opportunities to network including the Opening Reception, the Dali museum event, Beach Volleyball and, of course, the Beach Walk/Run for you early birds. There are no social formalities at NACRW, so please do not be shy about participating! I want to thank all of our workshop sponsors. Without their generosity, we simply could not host the Opening Reception and the Dali museum visit.

New this year is the NACRW Excellence Award sponsored by FLAG Works, Inc. This year the award focuses on Excellence in Sample Preparation. I am pleased to announce co-awardees Michelangelo Anastassiades and Steven Lehotay for the development, validation and implementation of the Quick, Easy, Cheap, Effective, Rugged and Safe (QuEChERS) as a sample preparation procedure for the analysis of pesticides and other chemical contaminants in food and agricultural matrices. Their work, efforts and approachability have been greatly impactful even beyond residue testing and we are honored to have both awardees at the workshop to address and inspire us.

I would like to thank the exhibitors and sponsors for participating in the workshop. Your partnership in our efforts has allowed us to offer a world class workshop while maintaining affordability for attendees. I need to thank my co-workers who support me and NACRW wholeheartedly. I would also like to thank all of the volunteers of the Organizing Committee, Program Committee, especially Jon Wong and Jian Wang, and Executive Director, Teri Besse, for the hard work and dedication. It has been my pleasure to serve with such caring and conscientious individuals.

Please enjoy!

Julie Kowalski, 2015 Organizing Committee President
Brad Barrett, Loretta Fourrier, and Joan Stevens (2015 Organizing Committee)
Jon Wong and Jian Wang (2015 Program Committee Co-Chairs) and the Program Committee members

FLAG Works, Inc. / North American Chemical Residue Workshop

Board of Directors

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GULF OF MEXICO

W S N E

BEACH

Cabana Hut

South Beach Lawn

Jacaranda Beach

Flying Bridge

Conclave

SeaBreeze Terrace

Poinciana

Salty's Beach Bar

ADULT POOL

Breck Deck North

Allamanda Bldg.

Jacaranda Bldg.

POOL & HOTTUB AREA

Bermudas

Beef 'O' Brady's

Breckenridge Bldg.

Pizza Hut Ice Cream

Body Works Salon

Hibiscus

Hibiscus

Horizons

West

East

Beakers Tropical Outfitters

Fitness Center

Garden Courtyard

Banyan Breezeway

Sharktooth Tavern

Portico

Pet Play Zone

Parking

VENDOR SEMINARS

Palm Court

POOL AREA

Tennis Courts

The Pavilion

Parking

South Parking

Island Ballroom

Grand Palm Colonnade CONFERENCE CENTER

Jacaranda Hall

Paddleboat Landings

Pirate Island

TAZ Activities

Cypress Villa

North Parking

EXHIBITS AND POSTERS

Lobby

Magnolia Offices

Parking

Parking

Admin Offices (5600)

HERTZ CAR RENTAL

REGISTRATION DESK AND MEETING ROOM

GULF BLVD.

TRADEWINDS SANDPIPER SUITES ▶

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

We would like to thank the following companies for their support of the 2015 NACRW

Platinum Sponsors

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ISLAND RESORTS ON ST. PETE BEACH
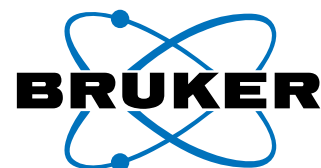
BRUKER

Confidence in your results comes from confidence in your technology

Whether you're testing for pesticides or other residues, establishing authenticity, or determining nutritional value, no one technology best meets every need. Bruker offers the broadest selection of high performance, easy-to-use, and expertly supported systems to overcome any food testing challenge. Our proven, extremely robust GC-Triple Quads, LC-Triple Quads and LC/GC-QqTOFs are designed for the food testing market to ensure absolute confidence in your results.

- GC-Triple Quadrupole
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- LC/GC-QqTOF

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2015 - 52ⁿᵈ ANNUAL NORTH AMERICAN CHEMICAL RESIDUE WORKSHOP

MEETING AT A GLANCE

Saturday, July 18, 2015

8:30 am – 4:30 pm	Short Course: Ana Lozano and Eric Verdon	Jasmine/Palm
	Pesticide and Veterinary Drug Residues: Method Validation and Routine Quality Control	

Sunday, July 19, 2015

8:00 am – 4:00 pm	Short Course: Ana Lozano and Eric Verdon	Jasmine/Palm
	Pesticide and Veterinary Drug Residues: Method Validation and Routine Quality Control	
1:00 – 5:00 pm	Exhibitor Setup	Pavilion
2:00 – 6:00 pm	Registration	Grand Palm Colonnade
4:00 – 5:30 pm	FDA/State Forum - government employees only	Sabal/Sawgrass
5:30 – 6:00 pm	Moderator and Volunteer Training	Ballrooms
6:15 – 7:15 pm	Vendor Seminar- Restek	Sawyer Key/Tarpon Key
7:30 – 9:30 pm	Welcome Reception	Pavilion

Monday, July 20, 2015

All Day	Registration	Grand Palm Colonnade
7:00 – 10:00 am	Poster Set Up	Pavilion
7:30 – 8:00 am	Early Morning Coffee	Grand Palm Colonnade
7:00 – 8:00 am	Vendor Seminar - Waters Corporation	Sawyer Key/Tarpon Key
8:10 – 8:20 am	Opening Remarks	Ballrooms
8:20 – 9:20 am	NACRW Excellence Award Presentation	Ballrooms
9:25 – 11:05 am	Difficult Residues and Difficult Matrices	Ballrooms
11:05 – 1:10 pm	Exhibition and Poster Opening	Pavilion
11:05 – noon	Poster Session (authors for odd #s)	Pavilion
12:10 – 1:10 pm	Vendor Seminar – Bruker Daltonics	Sawyer Key/Tarpon Key
1:15 – 2:55 pm	Freshwater and Marine Aquatic Toxins	Ballrooms
2:55 – 3:55 pm	BREAK (Exhibition & Posters)	Pavilion
2:55 – 3:55 pm	Poster Session (authors for even #s)	Pavilion
4:00 – 5:15 pm	Analysis Food Fraud/Adulteration	Ballrooms
6:00 pm	Social Event – Dali Museum	

Tuesday July 21, 2015

All Day	Registration	Grand Palm Colonnade
All Day	Exhibition & Posters	Pavilion
7:30 – 8:00 am	Early Morning Coffee	Pavilion
7:00 – 8:00 am	Vendor Seminar- SCIEX	Sawyer Key/Tarpon Key
8:15 – 9:55 am	General Topics	Ballrooms
10:00 – 12:55 pm	BREAK (Exhibition & Posters)	Pavilion
10:00 – 11:00 am	Poster Session (authors for even #s)	Pavilion
11:55 – 12:55 pm	Vendor Seminar- UCT, LLC	Tarpon Key
1:00 – 2:40 pm	Emerging Chemical Contaminants	Ballrooms
2:40 – 3:40 pm	BREAK (Exhibition & Posters)	Pavilion
2:40 – 3:40 pm	Poster Session (authors for odd #s)	Pavilion
3:45 – 5:25 pm	High Resolution Mass Spectrometry	Ballrooms
5:30 – 6:45 pm	Mass Spectrometry Forum	Ballrooms
6:50 – 7:30 pm	Organizing Committee Meeting - open to all attendees	Ballrooms
8:00 pm →	Beach Volleyball Game	on the Beach

MEETING AT A GLANCE

Wednesday, July 22, 2015

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	**Vendor Seminar- Thermo Scientific**	**Tarpon Key**
8:30 – 10:50 am	**Advanced Sample Comminution Techniques:**	**Ballrooms**
	The Future of Sample Processing in Residue Analytical Methods	
11:00 – noon	**BREAK (Exhibition & Posters)**	**Pavilion**
12:00 – 1:00 pm	**Vendor Seminar – Agilent Technologies**	**Tarpon Key**
1:05 – 2:45 pm	**Updates from Federal and State Regulatory Laboratories**	**Ballrooms**
2:45 – 3:30 pm	**BREAK**	**Grand Palm Colonnade**
3:00 – 3:30 pm	**AOAC Contaminants Community**	**Ballrooms**
	Pesticide Subgroup Introduction - open to all attendees	
3:30 – 5:10 pm	**Food Safety Issues**	**Ballrooms**
5:15 – 6:00 pm	**Poster Awards and Closing**	**Ballrooms**

Thursday, July 23, 2015
User Meetings

7:30 – 9:30 am	**SCIEX**	**Banyan**
10:30 – 12:30 pm	**Waters Corporation**	**Banyan**
10:30 – 12:30 pm	**Agilent**	**Citrus**
10:30 – 12:30 pm	**Thermo Scientific**	**Glades**

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 11:05 am – noon on Monday and 2:40 - 3:40 pm on Tuesday
Poster Session B (even#) authors must be at their posters from 2:55 pm – 3:55 pm on Monday and 10:00 - 11:00 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 8:30 pm and cash bar 8:30 to 9:30 pm
Monday - 11:00 am - 5:00 pm
Tuesday - 7:30 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. 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 23rd, 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 2015 NACRW. Please inform Jian Wang, 2015 Program Co-Chair (jian.wang@inspection.gc.ca), by August 31, 2015 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, 2015.

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

EXHIBITS AND POSTER SESSIONS
Location: Pavilion

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| 7 EPL Bio Analytical Services | 10 Shimadzu Scientific Instruments, Inc. | 20 LECO Corporation | 23 Pickering Laboratories | 33 Omni International | 36 Quantum Analytics |

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| 6 | 11 AccuStandard Inc. | | | 32 Biotage | 37 VUV Analytics, Inc. |

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FOOD & BEVERAGE

| 5 Advion | 12 Phenomenex | | | 31 Thermo Scientific | 38 Peak Scientific |

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8'   8'   8'   8'

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| 2 Teledyne Tekmar | 15 Agilent Technologies | 18 Bruker Daltonics | 25 Thomson Instrument Company | 28 Waters Corporation | 41 ITSP Solutions Inc |

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EXIT EXIT ENTRANCE EXIT EXIT

Exhibitors

AccuStandard, Inc
Booth #11
www.accustandard.com

Adpen Laboratories, Inc.
Booth #4
www.adpen.com

Advion
Booth #5
www.expressioncms.com

Agilent Technologies
Booths #15-16
www.agilent.com/chem

Biotage
Booth #32
www.biotage.com

Bruker Daltonics
Booth #18
www.bruker.com

Cambridge Isotope Laboratories, Inc.
Booth # 29
www.isotope.com

Chem Service, Inc.
Booth # 14
www.chemservice.com

Crescent Chemical Co., Inc.
Booth # 1
www.creschem.com

EPL Bio Analytical Services
Booth # 7
www.eplbas.com

GERSTEL, Inc.
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www.gerstelus.com

Glas-Col, LLC
Booth # 42
www.glascol.com

Horizon Technology Inc.
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ITSP Solutions Inc.
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LECO Corporation
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o2si Smart Solutions
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Peak Scientific
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Pickering Laboratories
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Quantum Analytics
Booth # 36
www.LQA.com

Restek
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www.restek.com

SCIEX
Booth # 26
www.sciex.com

Shimadzu Scientific Instruments, Inc.
Booths # 9-10
www.ssi.shimadzu.com

SPEX SamplePrep LLC
Booth # 8
www.spexsampleprep.com/

Supelco
Booth # 22
www.sigmaaldrich.com/analytical

Teledyne Tekmar
Booth # 2
www.teledynetekmar.com

Thermo Scientific
Booths # 30-31
www.thermoscientific.com

Thomson Instrument Company
Booth # 25
ww.htslabs.com

UCT, LLC
Booth # 17
www.unitedchem.com

ULTRA Scientific
Booth # 39
www.ultrasci.com

USDA, FSIS, OPHS, LQAS
Booth # 35
www.fsis.usda.gov

VUV Analytics, Inc.
Booth # 37
www.vuvanalytics.com

Waters Corporation
Booths # 27-28
www.waters.com

SHORT COURSE

Saturday, July 18, 2015 8:30 am to 4:30 pm
Sunday, July 19, 2015   8:00 am to 4:00 pm

Location: Jasmine/Palm Rooms

PRE-REGISTRATION IS REQUIRED.

Pesticide and Veterinary Drug Residues: Method Validation and Routine Quality Controls

Instructors:

Ana Lozano, European Union Reference Laboratory for Pesticide Residues in Fruit and Vegetable

and

Dr. Eric Verdon, French Agency for Food, Environmental and Occupational Health Safety and European Union Reference Laboratory for Antimicrobial and Dye Residues in Food

Method validation is essential and important to ensure the quality of data in an analytical chemistry laboratory. It is the process to demonstrate that a test method is capable of providing reliable data suitable for a specific application. This two day course provides a comprehensive overview and the fundamentals and execution of method validation procedures, quality assurance, and quality control as they pertain to pesticides and veterinary drugs. Attendees will obtain the knowledge to plan and execute method validation procedures for pesticides and veterinary drugs in food and animal feed. The course will cover elements required during the method validation process as well as the maintenance of established quality control protocols.

The course is designed for individuals who perform, supervise, manage and audit the validation of methods to ensure that data quality objectives are met. The course is also applicable to those who perform method validation as part of their occupational responsibilities.

- Topics to be covered (and additional topics will be covered if time permits) from the course include:
- Definitions and principles of validation parameters including LOD, LOQ, precision, accuracy, system suitability, reproducibility, ruggedness and robustness
- Approaches to Single and multi-analyte validation procedures
- EU and CODEX requirements and priorities and Harmonization
- Method Development and Optimization by Example and Case Studies such as using GC or LC-MS (MS/MS, HRMS) in Pesticides and Veterinary Drugs
- Representative matrices and analytes for multianalyte screening
- Differences and similarities of validation between different chemical classes (i.e., pesticides vs veterinary drugs)
- Statistical treatment of analytical data

Attendees will increase their knowledge and gain expertise in performing method validation protocols and acquire insight to address difficult and complex issues and tasks that involving validation.

EPRW
2016 Limassol

CATCH THE LEADING PESTICIDE MEETING OF 2016!!

The Pesticide Residues Laboratory of the State General Laboratory (SGL) of the Ministry of Health has the great pleasure and honor to invite you to the

EPRW 2016

11th European Pesticide Residue Workshop

MAY
**24 – 27
Limassol
Cyprus**

Join us and have the opportunity to participate
at the leading European meeting for the presentation
and discussion of the latest concepts and developments
in the field of pesticide residues in food and drink.

General topics :

- Development and Application of Analytical Methods for Pesticide Residues
- Analytical challenges caused by special matrices and analytes
- Monitoring, Risk Assessment and Regulatory Issues of Pesticides
- Quality Assurance / Quality Control / Accreditation

✓ *Talks from top scientists in the field of pesticides*
✓ *Vendor exhibits and seminars*
✓ *Exchange of information and experience with people of the relevant sectors*

Visit **www.eprw2016.com** for more information.

**We look forward in welcoming all of you to
Limassol for EPRW 2016!**

VENDOR SEMINARS

Food and beverage provided by each company

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

V-1 Sunday Evening, July 19, 2015, 6:15 p.m. to 7:15 p.m.   RESTEK
Location: Sawyer Key and Tarpon Key

Shoot-and-Dilute GC-MS/MS for the Analysis of Pesticides in Food

Jonathan Keim, Jack Cochran, Julie Kowalski
Restek Corporation, 110 Benner Circle, Bellefonte, PA 16823, USA; jonathan.keim@restek.com

Shoot-and-Dilute GC-MS/MS uses split injection paired with a very sensitive triple-quadrupole mass spectrometer to alleviate matrix-related issues occurring at the GC inlet and column. There are well-known problems associated with splitless injection of dirty samples (e.g. QuEChERS extracts), most notably compound degradation and the loss of relatively active and involatile pesticides. These issues lead to inaccurate quantification, and in some cases, completely missing the pesticide of interest. GC inlet problems can occur very quickly with real samples, sometimes with a single splitless injection of a particularly dirty sample. This leads to time-consuming inlet and column maintenance to restore instrument performance.

V-2 Monday, July 20, 2015, 7:00 to 8:00 am   WATERS CORPORATION
Location: Sawyer Key and Tarpon Key

Increasing Specificity of Accurate Mass HRMS Screening Experiments using Ion Mobility

Gareth Cleland, Waters Corporation, 34 Maple Street, Milford, MA; USA and Simon Hird, Waters Limited, Stamford Avenue, Altrincham Road, Wilmslow, UK

The use of HRMS and a non-targeted, data independent dataset for multi-residue analyses is appealing for several reasons:
•   It is possible to screen for an unlimited number of compounds due to data being acquired in full scan, non-targeted fashion.
•   Increased selectivity can be obtained for targeted compounds compared to SIR or MRM acquisitions.
•   Collection of a non-targeted dataset allows for historical data review.
•   Screening for unknown compounds of interest is possible.

When incorporated with high resolution mass spectrometry, ion mobility (IM) can reduce sample complexity, whilst increasing specificity. It offers an additional analytical measurement, collisional cross section (CCS), which efficiently reduces false detection rates, even when less stringent tolerances are applied to the other screening parameters.

The key to the successful implementation for routine analysis relies on the capability of accurate detection of residues at low concentrations with an acceptable level of false positive and false negative detects. In this work we will present a novel way to use IM-MS routinely in screening experiments and illustrate how robust CCS values can efficiently reduce false detection rates.

V-3 Monday, July 20, 2015, 12:10 to 1:10 pm   BRUKER DALTONICS
Location: Sawyer Key and Tarpon Key

Mass Spectrometric Strategies for Accurate Screening and Quantitation of Chemical Residues

Joe Anacleto – VP Applied Markets Business and Carsten Baessmann- Applications Development, Applied Markets, Bruker Daltonics, Billerica, MA; USA

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 presentation 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. Real world examples will 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.

V-4 Tuesday, July 21, 2015, 7:00 to 8:00 am   SCIEX
Location: Sawyer Key and Tarpon Key

Tips to Reduce Matrix Effects, Increase Throughput, and Decrease Data Processing Time for Routine Food Testing

André Schreiber, Applications Manager Food & Environment Markets, SCIEX, Concord, ON (Canada)

Mass spec is very common in routine food testing, but labs are still facing challenges to meet the ever-growing demands of food safety testing. Complex foods continue to produce matrix effects that can create highly unreliable quantitative results. Many chemical compounds must be analyzed by single-residue methods, adding significant time for analysis when multiple methods must be run on each sample. And, with multi-residue methods easily logging 100s of compounds, data processing across large batches of samples can take hours or even days. It's time to address these challenges head-on. This seminar will present new LC-MS/MS tools, tips, and workflows designed to help food testing labs overcome these and other every-day obstacles. A preview to future innovations from SCIEX will also be presented.

V-5 **Tuesday, July 21, 2015 11:55 to 12:55 pm** **UCT, LLC**
Location: Sawyer Key and Tarpon Key

Veterinary Drug Residue Analysis: Using Polymeric SPE and UHPLC-MS/MS to Develop a Multi-Class, Multi-Residue Method in Milk

Brian Kinsella, UCT, Bristol, PA, USA

Veterinary drugs are widely used in food-producing animals, including dairy animals. Milk is a popular food source that is consumed by a large portion of the population and is a staple for many toddlers and young children. To ensure food safety and prevent the unnecessary exposure of consumers to chemical contaminants it is important to test for veterinary drug residues in milk. The use of a multi-class, multi-residue (MMR) method is desirable, although this can be challenging due to the complexity of the sample matrix and the inclusion of a large number of drugs with diverse physicochemical properties. A MMR method should ideally be capable of extracting a wide range of drugs, reduce major matrix interferences, obtain good analyte recovery and achieve adequate limits of detection (LOD's). The latter requirement is important for milk as its MRLs are often lower than other biological matrices. The use of a generic sample preparation procedure, such as solid-phase extraction (SPE), is the most suitable approach for achieving these goals.

UHPLC-MS/MS is the detection system of choice for veterinary drugs as it allows rapid detection of trace-level residues in complex matrices. However, the diverse physicochemical properties of the veterinary drugs still pose challenges. Analytical conditions must be optimized to obtain adequate sensitivity of all the compounds as well as good retention and peak shape of problematic compounds.

This presentation will outline a method for the determination of multiple classes of veterinary drug residues in milk using a polymeric SPE cartridge and UHPLC-MS/MS analysis. The presentation will include a general overview of the analysis of veterinary drug residues in food, including major difficulties observed for certain drug classes and ways to overcome or mitigate them.

V-6 **Wednesday, July 22, 2015, 7:15 to 8:15 am** **THERMO SCIENTIFIC**
Location: Sawyer Key and Tarpon Key

Exploring Better Pesticide Residue Analysis Workflows

Katarzyna Banaszewski, Now Foods; Richard Fussell and Dipankar Ghosh, Thermo Fisher Scientific

Comprehensive screening, detection, identification and quantification of pesticide residues, irrespective of matrix, requires LC-MS and GC-MS based approaches, each using targeted and non-targeted acquisition.

Discover the new Thermo Scientific™ Pesticides Explorer Collection, a set of solutions based on Triple Quadrupole MS and Orbitrap™ technology, enabling laboratories to optimize start-up times, streamline

workflows and improve productivity. These workflows are specifically tailored to laboratories performing routine quantitation, targeted screening and non-targeted analysis of pesticide residues in food and enable users to easily obtain reliable, unambiguous, high quality LC-MS results.

We'll also demonstrate the excellent selectivity and sensitivity of GC-MS/MS in the analysis of 310 pesticides in botanical dietary supplements. Details of the method workflow will be discussed along with a verification procedure to determine method transferability from one sample type to another. Acquisition control and data processing were performed using the new Thermo Scientific™ Dionex™ Chromeleon™ 7.2 Chromatography Data System (CDS) software. The method performance exhibited applicability for routine pesticide screening in a wide variety of botanical dietary ingredients, particularly when combined with the simplified data acquisition and evaluation flow.

V-7 Wednesday, July 22, 2015, 12:00 to 1:00 pm   Agilent Technologies, Inc.
Location: Sawyer Key and Tarpon Key

Enhanced Matrix Removal: Next Generation Material for Improving the Analysis of Complex Samples

Derick Lucas, Limian Zhao, Bruce Richter, and Joan Stevens, Agilent Technologies, Inc., Wilmington, DE, USA

Analysis of complex matrices often requires extensive sample preparation to extract analytes of interest at the appropriate concentration, while removing unwanted matrix co-extractives. These co-extracted matrix components, such as lipids, can result in chromatographic interferences, ion suppression/enhancement in mass spectrometry, accumulation in chromatographic flow paths, and other performance hindering issues. This work demonstrates the benefits of using a new dispersive cleanup material with QuEChERS that dramatically reduces matrix co-extractives while maintaining excellent analytical accuracy and precision without additional steps, cost, or hardware. Data will be presented to demonstrate notable improvements on instrumental and chromatographic performance with cleaner sample matrices using the new material. Additionally, data will show the impact of superior cleanliness when conducting multi-residue analyte analysis in complex sample matrices using LC and GC. The ease of use, time and cost savings, minimal method development, and dramatically cleaner samples make this an attractive cleanup option for laboratories conducting trace analysis, especially in complex, fatty matrices.

2015 NACRW EXCELLENCE AWARDS

PRESENTED TO

Dr. Michelangelo Anastassiades, CVUA-Stuttgart, Germany

Dr. Michelangelo Anastassiades is currently the Head of EU Reference Laboratory for Pesticide Residues requiring Single Residue Methods. Dr Anastassiades activities include coordinating Network of National Reference Laboratories, method development and validation, development of Pesticide-related Databases, conduction of workshops and training courses for EU and developing country labs, organization of proficiency testing programs and technical assistance to DG-SANCO & EFSA. He has contributed to three books, published over 25 scientific publications, over 60 poster presentations and over 120 oral presentations. We are pleased the NACRW Excellence Award will be added to Dr. Anastassiades other achievements. His presentation is titled **Exploring the Limits of QuEChERS**.

Dr. Steven J. Lehotay, USDA-ARS, Wyndmoor, PA, USA

Dr. Steve Lehotay is a Lead Scientist with the Agricultural Research Service of the United States Department of Agriculture. Since 1992, scientific investigations and method development research have involved improvement in the analysis of pesticides, veterinary drugs, and other contaminants in food and environmental samples. Research has addressed all aspects of the analytical process, including sample preparation, cleanup, separations, detection, screening, quantification, identification/confirmation, and data processing using many types of analytical techniques applied in novel and useful ways. Dr. Lehotay has been invited to give lectures at more than 120 scientific meetings in more than 25 countries on 6 continents. He has authored and coauthored more than 130 scientific publications and more than 200 scientific abstracts. The NACRW Excellence Award will be added to Dr. Lehotay's numerous awards. We are pleased he will be presenting a reflective perspective in his talk titled **Streamlined Sample Preparation that Works So Well Needs a Catchy Name**.

2015 - 52nd Annual North American Chemical Residue Workshop

MEETING PROGRAM

Saturday, July 18, 2015

8:30 am – 4:30 pm **Short Course: Ana Lozano and Eric Verdon** **Jasmine/Palm**
Pesticide and Veterinary Drug Residues: Method Validation and Routine Quality Control

Sunday, July 19, 2015

8:00 am – 4:00 pm **Short Course: Ana Lozano and Eric Verdon** **Jasmine/Palm**
Pesticide and Veterinary Drug Residues: Method Validation and Routine Quality Control

1:00 – 5:00 pm	Exhibitor Setup	Pavilion
2:00 – 6:00 pm	Registration	Grand Palm Colonnade
4:00 – 5:30 pm	**FDA/State Forum** *(US States and Federal Employees only)*	Sabal/Sawgrass
5:30 – 6:00 pm	Moderator and Volunteer Training	Ballrooms

6:15 – 7:15 pm **Restek Evening Seminar** **Sawyer Key/Tarpon Key**
V-1 **Shoot-and-Dilute GC-MS/MS for the Analysis of Pesticides in Food**
Jonathan Keim, Jack Cochran, Julie Kowalski, Restek Corporation, Bellefonte, PA, USA

7:30 – 9:30 pm **Welcome Reception** **Pavilion**

Monday, July 20, 2015

All Day	Registration	Grand Palm Colonnade
7:00 – 10:00 am	Poster Set Up	Pavilion
7:00 – 8:00 am	Early Morning Coffee	Grand Palm Colonnade

7:00 – 8:00 am **Waters Corporation Breakfast Seminar** **Sawyer Key/Tarpon Key**
V-2 **Increasing Specificity of Accurate Mass HRMS Screening Experiments using Ion Mobility**
Gareth Cleland, Waters Corporation, Milford, MA, USA and Simon Hird, Waters Limited, Wilmslow, UK

8:10 – 8:20 am **Opening Remarks** **Ballrooms**
J.D. Warren, FLAGWORKS Chair

8:20 – 9:20 am **NACRW Excellence Award Presentation** **Ballrooms**
(60 min) **Julie Kowalski**, 2015 NACRW President

8:20 – 8:30 am Introduction and Presentation of Award

8:30 – 9:20 am Co-Recipients of the 2015 NACRW Excellence Award for Sample Preparation

Dr. Steven J. Lehotay, USDA-ARS, Wyndmoor, PA
A-1 **Streamlined Sample Preparation that Works So Well Needs a Catchy Name**

Dr. Michelangelo Anastassiades, CVUA-Stuttgart, Germany
A-2 **Exploring the Limits of QuEChERS**

9:25 – 11:05 am	**Difficult Residues and Difficult Matrices**	**Ballrooms**

Co-Chairs: **Yoko Johnson** and **Michael Filigenzi**

9:25 – 9:30 am Introduction

9:30 – 9:50 am **Brittany J. Holmes**, WSDA Chemical and Hop Laboratory, Yakima, WA, USA
O-1 **Development and Evaluation of a Multi-Class Analysis of Nine Halogenated Environmental Contaminants in Salmon by Liquid Chromatography-Tandem Mass Spectrometry**

9:55 – 10:15 am **Kuniyo Sugitate**, Agilent Technologies, Tokyo, Japan
O-2 **Applying Metabolomics Technique to Reveal Matrices in the Pesticide Residue Analysis of Foods**

10:20– 10:40 am **Yuhong Qin**, China Agricultural University, Beijing, China
O-3 **Utilization of Multiple Walled Carbon Nanotubes (MWCNTs) in d-SPE Cleanup and Multi-Plug-Filtration-Cleanup (m-PFC) Method Development**

10:45 – 11:05 am **Michael J. Hastings**, Dow AgroSciences, Indianapolis IN, USA
O-4 **Development of a Molecular Imprinted Polymer SPE and LC/MS/MS Method for the Analysis of Pyridine Herbicides in Compost Samples**

11:05 – 1:10 pm	**Exhibition and Poster Opening**	**Pavilion**
11:05 – noon	**Poster Session A (authors for odd #s)**	**Pavilion**

12:10 – 1:10 pm	**Bruker Daltonics Lunch Seminar**	**Sawyer Key/Tarpon Key**
V-3		

Mass Spectrometric Strategies for Accurate Screening and Quantitation of Chemical Residues
Joe Anacleto – VP Applied Markets Business and Carsten Baessmann – Applications Development, Applied Markets, Bruker Daltonics, Billerica, MA, USA

1:15 – 2:55 pm	**Freshwater and Marine Aquatic Toxins**	**Ballrooms**

Chair: **Marc Engel**   Moderator: **Walter Hammack**

1:15 – 1:20 pm Introduction

1:20 – 1:40 pm **Pearse McCarron**, National Research Council, Halifax, Nova Scotia, Canada
O-5 **Addressing the Challenges in Marine and Freshwater Toxin Analysis**

1:45 – 2:05 pm **Ryan Farmer**, Beagle Bioproducts, Inc., Columbus, OH, USA
O-6 **Detection of Microcystins: Does ADDA Add Up?**

2:10 – 2:30 pm **Jonathan Deeds**, US FDA/CFSAN, College Park, MD, USA
O-7 **Management of Human Exposure to Marine Natural Toxins from Contaminated Seafood**

2:35 – 2:55 pm **Leanne J. Flewelling**, Florida Fish and Wildlife, Conservation Commission, Fish and Wildlife Research Institute, St. Petersburg, FL, USA
O-8 **Harmful Effects of Marine Algal Toxins on Aquatic Wildlife**

2:55 – 3:55 pm	**BREAK (Exhibition & Posters)**	**Pavilion**
2:55 – 3:55 pm	**Poster Session B (authors for even #s)**	**Pavilion**

4:00 – 5:15 pm	**Analysis Food Fraud/Adulteration**	**Ballrooms**

Chair: **Katerina Mastovska**

4:00 – 4:05 pm Introduction

4:05 – 4:25 pm **O-9**	**Lukas Vaclavik**, Covance Laboratories, UK **Screening of Phosphodiesterase Type 5 Inhibitors in Dietary Supplements Using Liquid Chromatography/Quadrupole-Orbital Ion Trap Mass Spectrometry**
4:30 – 4:50 pm **O-10**	**Kurt-Peter Raezke**, Intertek Food Service, GmbH, Bremen, Germany **Honey Adulteration: How to Detect the Unknown**
4:55 – 5:15 pm **O-11**	**Patricia L. Atkins**, SPEX CertiPrep, Metuchen, NJ, USA **Estimation of the Adulteration, Counterfeiting and Contamination of Spices, Botanical Products, and Supplements by Detection of Heavy Metals and Potential Adulteration Compounds using ICP-OES and ICP-MS**
6:00 pm - 10 pm	**Social Event – Dali Museum**

Tuesday July 21, 2015

All Day	Registration	Grand Palm Colonnade
All Day	**Exhibition & Posters**	**Pavilion**
7:00 – 8:00 am	**Early Morning Coffee**	**Pavilion**
7:00 – 8:00 am **V-4**	**SCIEX Breakfast Seminar** **Tips to Reduce Matrix Effects, Increase Throughput, and Decrease Data Processing Time for Routine Food Testing** André Schreiber, Applications Manager Food & Environment Markets, SCIEX, Concord, Ontario, Canada	**Sawyer Key/Tarpon Key**
8:15 – 9:55 am	**General Topics** Chair: **Sherri Turnipseed**	**Ballrooms**
8:15 - 8:20 am	Introduction	
8:20 - 8:40 am **O-12**	**Eric Verdon**, ANSES, Laboratory of Fougères, European Union Reference Laboratory and National Reference Laboratory for Veterinary Drug Residues in Food from Animal Origin, France **New Approaches for the (Broad) Screening of Veterinary Drugs by Full Scan Accurate Mass Determination**	
8:45 - 9:05 am **O-13**	**Fadi Aldeek**, Florida Department of Agriculture and Consumer Services, Tallahassee, FL, USA **LC-MS/MS Method for Determination and Quantitation of Penicillin G and its Metabolites in Citrus Fruits Infected with Huanglongbing**	
9:10 - 9:30 am **O-14**	**Simon Hird**, Waters Corporation, Wilmslow, Cheshire, UK **Quantification of Regulated and Non-Regulated Lipophilic Marine Biotoxins by LC-MS/MS**	
9:35 – 9:55 am **O-15**	**Tao Li**, US FDA-San Francisco, CA, USA **False-Positive and False-Negative Concerns with LC-QQQ- in Production Lab: Risk Assessment**	
10:00 – 12:55 pm	**BREAK (Exhibition & Posters)**	**Pavilion**
10:00 – 11:00 am	**Poster Session B (authors for even #s)**	**Pavilion**

11:55 – 12:55 pm	**UCT, LLC Lunch Seminar**	**Sawyer Key/Tarpon Key**
V-5	**Veterinary Drug Residue Analysis: Using Polymeric SPE and UHPLC-MS/MS to Develop a Multi-Class, Multi-Residue Method in Milk**	
	Brian Kinsella, UCT, Bristol, PA, USA	

1:00 – 2:40 pm	**Emerging Chemical Contaminants**	**Ballrooms**
	Chair: **Paul Yang** Moderator: **Kai Zhang**	

1:00 – 1:05 pm	Introduction

1:05 – 1:25 pm	**Ruth Marfil-Vega**, American Water, Belleville, IL, USA
O-16	**Evaluation of Emerging and Unregulated Drinking Water Contaminants and the Impact of Operations at American Water Facilities**

1:30 - 1:50 pm	**Tarun Anumol**, Agilent Technologies, Wilmington, DE, USA
O-17	**Comparison of Analytical Methodologies for Analysis of Emerging Organic Contaminants in Water**

1:55 – 2:15 pm	**Jonathan D. Byer**, LECO Corporation, Saint Joseph, MI, USA
O-18	**Non-Target Analysis of E-Waste Samples from China Using GCxGC-HRTOFMS**

2:20 – 2:40 pm	**Thinh Duc Nguyen**, Institute of Public Health, Ho Chi Minh City, Vietnam
O-19	**Antibiotic Residue Monitoring for Animal Food in Ho Chi Minh City, Vietnam**

2:40 – 3:40 pm	**BREAK (Exhibition & Posters)**	**Pavilion**
2:40 – 3:40 pm	**Poster Session A (authors for odd #s)**	**Pavilion**

3:45 -5:25	**High Resolution Mass Spectrometry**	**Ballrooms**
	Co-Chairs: Walter Hammack and Mark Crosswhite	

3:45 – 3:50 pm	Introduction

3:50 – 4:10 pm	**Michael S. Filigenzi**, California Animal Health and Food Safety Laboratory, Davis, CA, USA
O-20	**Recent Applications of High Resolution Mass Spectrometry in Veterinary Toxicology**

4:15 – 4:35 pm	**Mark R. Crosswhite,** Florida Department of Agriculture and Consumer Services, Tallahassee, FL, USA
O-21	**Understanding Where the Specificity Afforded by "True" MS/MS is Fully Diminished Due to Increased Isolation Ranges Commonly Used in "Quasi-MS/MS" High Resolution Accurate Mass Measurements**

4:40 – 5:00 pm	**Dominic Roberts,** Thermo Fisher Scientific, Runcorn, UK
O-22	**Potential of GC Orbitrap MS Technology for the Analysis of Pesticide Residues in Food and Feed**

5:05 – 5:25 pm	**Ann M. Knolhoff**, US FDA/CFSAN, College Park, MD, USA
O-23	**Determination of HR-MS Data Quality for Non-Targeted Screening of Food Matrices**

5:30 – 6:45 pm	**Mass Spectrometry Forum**	**Ballrooms**
	Coordinators: Walter Hammack and Mark Crosswhite	

6:50 – 7:30 pm	**Organizing Committee Meeting** – *open to all attendees*	**Ballrooms**

8:00 pm →	**Night Beach Volleyball Game**	On the Beach

Wednesday, July 22, 2015

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

7:15 – 8:15 am
V-6
    Thermo Scientific Breakfast Seminar   **Sawyer Key/Tarpon Key**
Exploring Better Pesticide Residue Analysis Workflows
Katarzyna Banaszewski, Now Foods; Richard Fussell and
Dipankar Ghosh, Thermo Fisher Scientific

8:30 – 10:50 am   **Advanced Sample Comminution Techniques:**   **Ballrooms**
The Future of Sample Processing in Residue
Analytical Methods
Co- Chairs: Leah Riter and Jo Marie Cook

8:30 – 8:35 am   Introduction

8:35 - 8:55 am
O-24
Árpád Ambrus, National Food Chain Safety Office (retired), Budapest, HUNGARY
Contribution of Sample Processing to Variability and Accuracy of Measured Residues

9:00 - 9:20 am
O-25
Jo Marie Cook, Florida Department of Agriculture and Consumer Services, Tallahassee, FL, USA
Theory of Sampling Guide to Quality Sample Processing

9:25 -9:45 am
O-26
Leah S. Riter, Monsanto, St Louis, MO, USA
Cryogenic Milling: An Enabling Technology for High Throughput Residue Sample Preparation

9:50-10:20 am
O-27
Kari J. Lynn, Dow AgroSciences, Indianapolis IN, USA
Assessment of Cryomilling Sample Processing of Plant and Animal Tissues for High Throughput Residue Analysis

10:20 – 10:50 am   **Discussion Panel**

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

12:00 – 1:00 pm
V-7
Agilent Technologies Lunch Seminar   **Sawyer Key/Tarpon Key**
Enhanced Matrix Removal: Next Generation Material for Improving the Analysis of Complex Samples
Derick Lucas, Limian Zhao, Bruce Richter, and Joan Stevens, Agilent Technologies, Inc., Wilmington, DE, USA

1:05 – 2:45 pm   **Updates from Federal and State Regulatory Laboratories**   **Ballrooms**
Co-Chairs: Steven C. Moser and Ping Wan

1:05 – 1:10 pm   Introduction

1:10 – 1:30 pm
O-28
Michael R. Curry, Georgia Department of Agriculture, Tifton, GA, USA
Level of 2,4-D or Dicamba Residue Found in Cucurbit Fruit from a Simulated Drift Scenario

1:35 – 1:55 pm **O-29**	**Lawrence B. Zintek**, US EPA, Chicago, IL, USA **Quick Method for the Analysis of Select Midwestern United States Applied Pesticides in Surface Water from Agricultural Run-off using a Simple Sample Preparation Followed by UPLC/MS/MS Analysis**
2:00 – 2:20 pm **O-30**	**Kai Zhang**, US FDA/CFSAN, College Park, MD, USA **Development of LC-MS Based Multi-Mycotoxin Methods for U.S. FDA Compliance Testing and Surveillance**
2:25 – 2:45 pm **O-31**	**Chris Sack**, US FDA-Kansas City, KS, USA **FDA's Total Diet Study Program – An Update**

2:45 – 3:30 pm	**BREAK**	**Grand Palm Colonnade**
3:00 – 3:30 pm	**AOAC Contaminants Community** **Pesticide Subgroup Introduction** - *open to all attendees* **AOAC Pesticide Subgroup Chairs: Steven C. Moser and Ping Wan**	**Ballrooms**
3:30 – 5:10 pm	**Food Safety Issues** **Co-Chairs: Sherry Garris and Brian Eitzer**	**Ballrooms**

3:30 – 3:35 pm	Introduction
3:35 – 3:55 pm **O-32**	**Ana Lozano**, European Union Reference Laboratory for Pesticide Residues in Fruit & Vegetables, SPAIN **Matrix Effects within the Main Multiresidue Methods in Fruits and Vegetables**
4:00 – 4:20 pm **O-33**	**Chris Pappas**, USDA Agricultural Marketing Service, Washington, DC, USA **Pesticide Data Program Sampling – Obtaining a Representative Sample**
4:25 – 4:45 pm **O-34**	**Paul H. Reibach**, Smithers Viscient, Wareham, MA, USA **Plant and Animal Pesticide Method Radiovalidation Studies: Implications for Methodologies Used for Subsequent Food Safety Testing**
4:50 – 5:10 pm **O-35**	**Johannes Corley**, Food and Environmental Protection Section, Joint FAO/IAEA Division of Nuclear Techniques in Food and Agriculture, Vienna, AUSTRIA **Food Safety and Security in the Modern World, Problems and Challenges Facing Scientists in Building Consumer Confidence in the Safety of our Food Supply. How do Nuclear Applications Fit into Food Security?**

5:15 – 6:00 pm	**Poster Awards and Closing**	**Ballrooms**

Thursday, July 23, 2015

User Meetings

7:30 – 9:30 am	**SCIEX**	**Banyan**
10:30 – 12:30 pm	**Waters Corporation**	**Banyan**
10:30 – 12:30 pm	**Agilent**	**Citrus**
10:30 – 12:30 pm	**Thermo Scientific**	**Glades**

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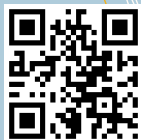
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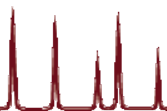
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POSTERS

P-1   **Next Generation Matrix Removal Materials for Multi-Analyte Analysis**
Derick Lucas, et al.; Agilent Technologies, Inc., Wilmington, DE, USA

P-2   **Optimize Food Analysis with Miniaturized QuEChERS and an Ultra-Efficient Triple Quadrupole GC/MS**
Joan Stevens, Agilent Technologies Inc., Wilmington, DE 19808, USA and Melissa Churley, Agilent Technologies Inc., Santa Clara, CA, USA

P-3   **Improving the Robustness of Daily Instrument Analysis through Enhanced Sample Matrix Removal**
Limian Zhao, et al.; Agilent Technologies, Inc., Wilmington, DE, USA

P-4   **Screening for Hundreds of Pesticides in Fruits and Vegetables using a High Resolution Accurate Mass GC/Q-TOF with an Exact Mass Pesticide Library**
Philip L. Wylie, et al.; Agilent Technologies, Wilmington, DE, USA

P-5   **Identification of Emerging Organic Contaminants in Water by Liquid Chromatography Time-of-Flight Mass Spectrometry**
Jerry Zweigenbaum & Tarun Anumol, Agilent Technologies, Inc., Wilmington, DE, USA

P-6   **LC/QTOF MS Determination of Pyrrolizidine Alkaloids in Dietary Supplements and Botanicals**
Jerry Zweigenbaum, et al.; Agilent Technologies, Inc., Wilmington, DE, USA

P-7   **Improvements in the QuEChERS Method for Multi-residue Analysis of Pesticides in Tobacco**
Joan Stevens, et al.; Agilent Technologies, Inc., Wilmington, DE, USA

P-8   **Determination of Imidazolinone Herbicides in Food of Plant Origin, By an Automated Extraction Platform**
Tyler Trent and Tom Hartlein, Teledyne Tekmar ,Mason, OH, USA

P-9   **Tetracycline and Fluoroquinolone Residues in Meat, Chicken and Fish using Automation and New Consumables**
Chris Shevlin, et al.; Horizon Technology Salem, NH USA

P-10   **Statistical Determination of Stability from Two Multi-Component Pesticide Mixes – Part Two**
Kelly Dorweiler, General Mills/Medallion Laboratories, Golden Valley, MN, USA; and Jagdish Gurav, General Mills/Medallion Laboratories Powai, Mumbai, India

P-11   **Determination of Veterinary Drug Residues in Milk Using SPE and UHPLC-MS/MS with a Polyaromatic HPLC Column**
Brian Kinsella, UCT Inc., 2731 Bartram Road; Bristol, PA 19007, USA

P-12   **Analysis of Polychlorinated Terphenyls by GC-EI Triple Quadrupole Mass Spectrometry**
Louis Maljers, Gordon van 't Slot, Bruker Daltonics, Bremen, Germany

P-13   **Screening and Quantitation of About 200 Pesticides in Honey by an Integrated On-Line Extraction UHPLC-MS/MS System**
Zicheng Yang and Louis Maljers, Bruker Daltonics Inc, Fremont, CA, USA

P-14   **Analysis of selected POPs with an atmospheric pressure chemical ionisation GC coupled to high-resolution QTOFMS**
Dorith Claes, et al.; Bruker Daltonik GmbH; Bremen; Germany

P-15   **Multi-target Pesticide Screening using Atmospheric Pressure Chemical Ionization GC Coupled to High-Resolution Q-TOF-MS**
Carsten Baessmann, et al.; Bruker Daltonik GmbH; Bremen; Germany

P-16   **Development and Evaluation of a New Software Tool for Confident Identification of Pesticides in Food Extracts using High Resolution QTOF Mass Spectrometry**
Carsten Baessmann, et al.; Bruker Daltonik GmbH; Bremen; Germany

P-17   **Automation of EPA Method 526: Determination of Selected Semi-Volatile Organic Compounds in Drinking Water by SPE and GC/MS.**
Leticia Brown, et al.; J2 Scientific, Columbia, MO, USA

P-18   **Organochlorine-Pesticides, Polychlorinated Biphenyls, and Polybrominated Diphenyl Ethers: An Automated Approach to Sample Preparation with Aquatic Biological Tissue Matrices.**
Michael Tanner, et al.; J2 Scientific, Columbia, MO, USA

P-20   **GC-MS/MS TQ Methods with Short and Long Run Times for Limited- or Extended-Scope Pesticide Residue Monitoring or Import Control Purposes, using the New Dutch ("NL-") Extraction Method**
Barbara A. Kiedrowska and André de Kok, NVWA - Netherlands Food and Consumer Product Safety Authority, Wageningen, The Netherlands

P-21   **Prediction of Dietary Possible Metabolic Residues in Body by Monitoring the Formation of Microbial Metabolites of Fungicide Cyazofamid by Soil Fungus *Cunninghamella elegans***
Hyeri Lee, et al.; National Institute of Environmental Research, Republic of Korea

P-22   **Establishment of Fast Multiresidue Pesticide Analysis for Water Samples by LC-MS/MS and GC-MS/MS**
Hyeri Lee, et al.; National Institute of Environmental Research, Republic of Korea

P-23   **The Use of High Resolution Hybrid Mass Spectrometry and Liquid Chromatography for the Screening, Quantification and Identification of 451 Pesticides in Fruits and Vegetables**
Jian Wang, et al.; Canadian Food Inspection Agency, Calgary, Alberta, Canada

P-24   **Simultaneous Analysis of the Flonicamid and its metabolites TFNG and TFNA in Rice and Soybean using LC-MS/MS**
Jeong-Han Kim, et al.; Seoul National University, Department of Agricultural Biotechnology, Republic of Korea

P-25   **Use of 13C isotope for Avoiding Linearity Problems in Time of Flight Mass Spectrometry**
Ana Lozano, et al.; European Union Reference Laboratory for Pesticide Residues in Fruit & Vegetables, University of Almeria, Agrifood Campus of International Excellence (ceiA3) Department of Hydrogeology and Analytical Chemistry, Almería, Spain

P-26   **GC-(NCI)-QTOF Approach with Automated Accurate Mass Data Processing for Determination of 70 Pesticides in Vegetables**
Ana Lozano, et al.; European Union Reference Laboratory for Pesticide Residues in Fruit & Vegetables, University of Almeria, Agrifood Campus of International Excellence (ceiA3) Department of Hydrogeology and Analytical Chemistry, Almería, Spain

P-27   **Multi-residue Determination of 69 Pesticides in Rice Straw Roughage by QuEChERS and HPLC-MS/MS**
Lijun Han, et al.; College of Science, China Agricultural University, Beijing, China

P-28   **Utilization of Multiple Walled Carbon Nanotubes (MWCNTs) in d-SPE Cleanup and Multi-Plug-Filtration-Cleanup (m-PFC) Method Development**
Yuhong Qin, et al.; Department of Applied Chemistry, China Agricultural University, Beijing, China

P-29   **Antibiotic Residue Monitoring for Meat and Egg in Ho Chi Minh City, Vietnam in 2014 - 2015**
Thinh Duc Nguyen, et al.; Institute of Public Health, Ho Chi Minh City, Vietnam

P-30   **Monitoring of Illegal Compounds, including Anti-impotence Drugs, Anti-obesity Drugs and their Analogues in Foods**
Jang-Duck Choi, et al.; Ministry of Food and Drug Safety, Republic of Korea

P-31   **Method for Determination of Pahs in Liver Samples of Green Sea Turtles (*Chelonia mydas)* Using HPLC-FLD**
Franz Zirena Vilca, et al.; Ecotoxicology Laboratory, Center of the Nuclear Energy in Agriculture CENA, University of São Paulo, Brazil

P-32   **Potential Role of PAHS in Green Sea Turtles Fibropapillomatosis**
Franz Zirena Vilca, et al.; Ecotoxicology Laboratory, Center of the Nuclear Energy in Agriculture CENA, University of São Paulo, Brazil

P-33   **Monitoring of PAHs in Liver Samples OF Green Sea Turtles (*Chelonia mydas*) With and Without Fibropapillomatosis Captured at Three Brazilian Feeding Areas**
Valdemar Luiz Tornisielo, et al.; Ecotoxicology Laboratory of the Center of the Nuclear Energy in Agriculture CENA. University of São Paulo, Brazil

P-34   **Determination of Organochlorine Pesticides in Organic Quinoa Grains**
Valdemar Luiz Tornisielo, et al.; Ecotoxicology Laboratory of the Center of the Nuclear Energy in Agriculture CENA. University of Sao Paulo, Brazil

P-35   **Antibiotic Residue Monitoring for Freshwater Products in Ho Chi Minh City and Thai Binh, Vietnam**
Kotaro Uchida, et al.; Osaka Prefectural Institute of Public Health, Osaka, Japan

P-36   **Risk Based Analysis of Chemical Hazards**
Grace Bandong, and Julie Hill, The National Food Laboratory, Livermore, CA, USA

P-37   **Analysis of 16 Antibiotics in Feedstuffs Using Ion Trap LC-MSⁿ**
Yoko S. Johnson, et al.; Minnesota Department of Agriculture, Saint Paul, MN, USA

P-38   **Developing an In-house Proficiency Test Sample for Aroclor 1254/1260 Mixture in Fish to Meet the ISO/IEC 17025:2005 Requirements**
Yoko S. Johnson, et al.; Minnesota Department of Agriculture, Saint Paul, MN, USA

P-39   **The Use of Liquid Chromatography/High Resolution Mass Spectrometry in the Analysis of Pesticide Residues on Produce**
Brian D. Eitzer, et al.; The Connecticut Agricultural Experiment Station, New Haven CT, USA

P-40   **Photodegradation of 2,6-dichloro-4-nitroaniline (DCNA) in Freshwater and Saltwater**
Emily Vebrosky and Kevin L. Armbrust, Department of Environmental Sciences, Louisiana State University, Baton Rouge, LA, USA

P-41   **Pesticide Sediment Partitioning and Exposure Modeling**
Brendan Marsh and Kevin L. Armbrust, Department of Environmental Sciences Louisiana State University, Baton Rouge, LA, USA

P-42   **Water Solubility Measurements of Atrazine and Fipronil, in Freshwater and Seawater**
Parichehr Saranjampour and Kevin L. Armbrust, School of the Coast and Environment, Department of Environmental Sciences; Louisiana State University, Baton Rouge, LA, USA

P-43   **A High-Throughput and Sensitive Method For Quantitation And Identification Of Chloramphenicol in Foods of Animal Origin Using UHPLC-MS/MS**
John Schmitz, et al.; Covance Nutritional Chemistry and Food Safety (NCFS), Madison, WI, USA

P-44   **Screening and Identification of Adulterants in Weight Loss Supplements by UHPLC and High-Resolution Accurate-Mass Detection**
John Schmitz, et al.; Covance Nutritional Chemistry and Food Safety (NCFS), Madison, WI, USA

P-45   **Highly Polar Pesticide Analysis in Food Samples by LC- MS/MS**
David R. Baker, et al.; Shimadzu Corporation, Manchester, United Kingdom

P-46   **Direct Determination of Trace Hormones in Drinking Water by Large Volume Injection at Sub ng/L Levels Using LC-MS/MS**
David R. Baker and Neil Loftus, Shimadzu Corporation, Manchester, United Kingdom

P-47   **Measurement of Antifungal Residues in a Treated Cannabis Crop**
Jeffrey H. Dahl, et al.; Shimadzu Scientific Instruments, Columbia, MD, USA

P-48   **Analysis of Veterinary Drug Residues in Livestock and Fishery Products**
Yuka Fujito and Jeffrey H. Dahl, Shimadzu Scientific Instruments, Columbia, MD, USA

P-49   **Pesticide Screening in the Non-Regulated Medical Marijuana Industry by GC-MS/MS**
Robert H. Clifford, et al.; Shimadzu Scientific Instruments, Columbia, MD, USA

P-50   **Pesticides Analysis by On-line SFE-SFC-MS/MS for Improved Sample Preparation, Analysis Time, and Sensitivity, Plus a Wider Range of Analyte Polarities Measured**
Robert H. Clifford, et al.; Shimadzu Scientific Instruments, Columbia, MD, USA

P-51   **A Simple and Rapid Extraction Method for Chlorinated Pesticides in Poultry Meat Using Solid Phase Extraction and GC/ECD**
Ramkumar Dhandapani, et al.; Phenomenex, Inc., Torrance, CA, USA

P-52   **High Resolution Accurate Mass (HRAM) Collision Energy Profile of Residues of Concern for Food Safety**
Huichen Stavros, o2si Smart Solutions, Charleston, SC, USA

P-53   **Cleanup of QuEChERS extracts using SBSE for LC/MS/MS determination of pesticides in food products**
Fred Foster, et al.; GERSTEL Inc., Linthicum, MD, USA

P-54   **Streamlined Sample Preparation Methodology to enable Higher Recovery, and minimize loss of Pesticides, Fungicides and Antibiotics by LC/MS or GC/MS.**
Lisa Wanders and Sam Ellis, Thomson Instrument Company, Oceanside, CA, USA

P-55   **The Analysis of Two Classes of Persistant Organic Pollutants in Challenging Edible Oil Samples**
Olga I. Shimelis, et al.; Sigma-Aldrich, Bellefonte, PA, USA

P-56   **Analysis of Bisphenol A in Milk and Canned Broths Using Molecularly Imprinted Polymer SPE and LC with Fluorescence Detection.**
Olga I. Shimelis, et al.; Sigma-Aldrich, Bellefonte, PA, USA

P-57   **Use of Graphitized Carbon Black and other Adsorbents for the Removal of Pigments during QuEChERS**
J. Patrick Myers, et al.; Sigma Aldrich/Supelco, Bellefonte, PA, USA

P-58   **Analysis of Antioxidants in foods and Dietary Supplements Using HPLC with Post-Column Derivatization**
Michael Gottschalk, Pickering Laboratories, Inc. Mountain View, CA, USA

P-59   **Modern State of GC-MS/MS Pesticide Analysis using Thermo Scientific™ TSQ™ 8000 Evo and Chromeleon™ 7.2 SR2 CDS**
Gail Harrison, et al.; Thermo Fisher Scientific, Austin, TX, USA

P-60   **Determination of meat authenticity using peptide biomarkers and high-resolution mass spectrometry**
Dipankar Ghosh, et al.; Thermo Fisher Scientific, San Jose, CA, USA

P-61   **EPA Method 557 Quantitation of Haloacetic Acids, Bromate and Dalapon in Drinking Water Using Ion Chromatography and Tandem Mass Spectrometry**
Jonathan Beck et al.; Thermo Fisher Scientific, San Jose, CA, USA

P-62   **Screening and Quantitation of Micropollutants from Sewage Water in the Process of Bank Filtration using UHPLC-HRMS**
Maciej Bromirski, et al; Thermo Fisher Scientific, Bremen, Germany

P-63   **Determination of a Single Methodology for the Analysis and Quantitation of Multi-class Veterinary Drugs in Different Animal Matrices used for Consumption**
Charles Yang, et al.; Thermo Fisher Scientific, San Jose, CA, USA

P-64   **Quantitative and Qualitative Confirmation of Pesticides in Beet Extract Using High Resolution Accurate Mass (HRAM) Mass Spectrometry**
Charles Yang, et al.; Thermo Fisher Scientific, San Jose, CA, USA

P-65   **Using Capillary IC with Suppressed Conductivity and Charge Detection to Profile Organic Acids in Juices and Beverages**
Todd Baker, et al.; ThermoFisher Scientific, Bannockburn, IL, USA

P-66   **The Use of XAD-2 Resin Packets For Passive Air Sampling of Chlorinated Pesticides**
William J. Luksemburg, et al.; Vista Analytical Laboratory, El Dorado Hills, CA, USA

P-67   **Analysis of EU 15+1 Priority Polycyclic Aromatic Hydrocarbons in Yerba Mate Tea Using Modified QuEChERS, Solid Phase Extraction and Gas Chromatography Time-of-Flight Mass Spectrometry**
Michelle Misselwitz,et al.; Restek, Bellefonte, PA, USA

P-68   **Optimization of Gas Chromatographic Parameters using Chromatographic Modelling Program for Halogenated Disinfection By-Products Analysis**
Paul Yang, et al.; Ontario Ministry of the Environment and Climate Change; Ontario, Canada

P-69   **Shoot-and-Dilute GC-ECD for Analysis of Problematic Pesticides**
Jack Cochran and Julie Kowalski, Restek, Bellefonte, PA, USA

P-70   **GC-MS/MS Method Development Strategies and Lessons Learned for Multiresidue Pesticides in Food Matrices**
Jack Cochran and Julie Kowalski, Restek, Bellefonte, PA, USA

P-71   **Liquid Chromatographic Methods for Pesticide Analysis and Cannabinoid Profiling in Cannabis**
Amanda Rigdon, et al.; Restek, Bellefonte, PA, USA

P-72   **Gas Chromatographic Methods for Analysis of Pesticides and Residual Solvents in Cannabis and Cannabis Concentrates**
Amanda Rigdon, et al.; Restek, Bellefonte, PA, USA

P-73   **Impact of Analytical Reporting Criteria on MRLs and Risk Assessment**
Carmen Tiu and Amy M. Phillips, Dow AgroSciences LLC, Indianapolis, IN, USA

P-74   **Quantitative Determination of a Pesticide and Its metabolites in Soil with Sonication Extraction**
Qian Li, et al.; Dow AgroSciences, Indianapolis, IN, USA

P-75   **Doing More with Less – The Advantages of Miniaturization and High-Throughput**
Louis C. Mayer, Syngenta Crop Protection, LLC, Greensboro, NC, USA

P-76   **Level of 2,4-D or Dicamba Residue Found in Cucurbit Fruit from a Simulated Drift Scenario using LC/MS/MS**
Jessica Flowers, et al.; Georgia Department of Agriculture, Tifton, GA, USA

P-77   **Sandwich Injection Method for Pesticide Analysis by Agilent 7000 GCMSMS System**
Ronald Francisco and Raymond Allum, Florida Department of Agriculture and Consumer Service, Division of Food Safety, Tallahassee, FL, USA

P-78   **Adduct Interferences Found in Large Pesticide Residue Screens Using High Resolution Mass Spectrometry**
Zaid Hamilton, et al.; Florida Department of Agriculture and Consumer Services, Tallahassee, FL, USA

P-79   **A Rapid Liquid Chromatography Determination of Formaldehyde in Cod**
Joseph M. Storey, et al.; Animal Drugs Research Center, U.S. Food and Drug Administration, Denver, CO, USA

P-80   **Strategies for the Application of HRMS for the Semi-targeted Analysis of Veterinary Drug Residues in Aquacultured Products**
Sherri B. Turnipseed, et al.; Animal Drugs Research Center, U.S. Food and Drug Administration, Denver, CO, USA

P-81   **Direct Determination of Glyphosate, Glufosinate, and AMPA in Milk by Liquid Chromatography/Tandem Mass Spectrometer**
Narong Chamkasem, et al.; SRL/FDA, Atlanta, GA, USA

P-82 **Direct Determination of Glyphosate, Glufosinate, and AMPA in Soybean and Corn by Liquid Chromatography/Tandem Mass Spectrometer**
Narong Chamkasem, et al.; SRL/FDA, Atlanta, GA, USA

P-83 **Rapid, New, Methods for the Analysis of 3-MCPD and 1,3 DCP in Soy Sauce**
Susan Genualdi, et al.; U.S. Food and Drug Administration, College Park, MD, USA

P-84 **Comparison of Multiple Methods for the Determination of Sulfite in *Allium* and *Brassica* Vegetables**
Katherine Robbins and Lowri de Jager, U.S. Food and Drug Administration, College Park, MD, USA

P-85 **GC-MS/MS Determination of Pesticides and Tobacco-Specific Nitrosamines (TSNAs) in Finished Cigarette Tobacco Using a Modified QuEChERS Method**
Mary B. Jones, et al.; US FDA Forensic Chemistry Center, Cincinnati, OH, USA

P-86 **Analysis of Acid Herbicides Using Modified QuEChERS with Fast Switching ESI+/ESI- LC-MS/MS Determination**
Chris Sack, et al.; U.S. Food and Drug Administration, Lenexa, KS, USA

P-87 **Validation Study of 204 Pesticides in Fruits and Vegetables by QuEChERS and LC-MS/MS**
Kelli Simon, et al.; U.S. Food and Drug Administration, Center for Food Safety and Applied Nutrition, College Park, MD, USA

P-88 **Evaluation of an Automated QuEChERS (Quick, Easy, Cheap, Effective, Rugged, Safe) Sample Preparation Workflow for Determination of Pesticides in Fresh Produce using LC-MS/MS**
Kelli Simon, et al.; U.S. Food and Drug Administration, Center for Food Safety and Applied Nutrition, College Park, MD, USA

P-89 **Comparison of Q-ToF and Q-Orbitrap HR/LCMS Platforms in a High-Throughput Regulatory Setting for Determination of ~1000 Pesticides, Toxins, Drugs and Other Unexpected Food Contaminants**
Greg E. Mercer, et al.; USFDA Pacific Regional Laboratory Northwest, Bothell, WA , USA

P-90 **Optimization of Solvent Polarity for Cleanup of QuEChERS Extracts and Recovery of Pesticides that Require Determination by Gas Chromatography**
Greg E. Mercer, et al.; USFDA Pacific Regional Laboratory Northwest, Bothell, WA , USA

P-91 **Multiresidue Pesticide Analysis of Teas using Ultrahigh Performance Liquid Chromatography coupled with Quadrupole-Orbitrap Mass Spectrometry**
Jon W. Wong, et al.; United States Food and Drug Administration, Center for Food Safety and Applied Nutrition, College Park, MD, USA

P-92 **Pesticides and Persistent Organic Pollutants in CAMELLIA *SINENSIS***
Douglas G. Hayward, et al.; United States Food and Drug Administration, Center for Food Safety and Applied Nutrition, College Park, MD, USA

P-93 **Broad Spectrum Pesticide Residue Analysis of Bee Pollen collected from the Northern Great Plains**
Alaa Kamel and Clint Otto, Analytical Chemistry Laboratory, Office of Pesticide Programs, US Environmental Protection Agency, Fort George G. Meade, MD, USA;

P-94 **Challenges for Analysis of Pyrethroid Residues: Overall Strategies and Solid Phase Micro-extraction (SPME) Case Study**
Del A. Koch, et al.; ABC Laboratories, Columbia, MO, USA

P-95 **The Development and Validation of a High-Performance (HP) Lateral Flow Immunoassay (LFIA) for the Rapid Screening of a Domoic Acid (DA) from Shellfish Extracts**
Jennifer Rice, et al.; Neogen Corporation, Lansing, MI, USA

P-96 **The Development and Validation of a High-Performance (HP) Lateral Flow Immunoassay (LFIA) for the rapid Screening of Okadaic Acid (OA) and Dinophysistoxins (DTXs) from Shellfish Extracts**
Jennifer Rice, et al.; Neogen Corporation, Lansing, MI, USA

P-97 **Authenticity of Spice: Detection and Quantification of Allergens in Spice using LC-MS**
Robert D. Voyksner, et al.; LCMS Limited and ImmunogenX, Durham, NC, USA

P-98   **Are Perfluorinated Compounds Leaching Into Our Food?**
Liesl Krone and André Schreiber, Granbury High School, Granbury, TX, USA

P-99   **Quantitation of the Pesticide 1080 (Sodium Fluoroacetate) in Milk and Infant Formula**
Farzad Pakdel, et al.; SCIEX, Redwood City, CA, USA

P-100   **The Use of LC-MS/MS for the Identification of Allergens in Spices**
Farzad Pakdel, et al.; SCIEX, Redwood City, CA, USA

P-101   **Identification, Quantitation and Confirmation of Pesticides in Food Samples using Advanced LC-MS/MS Techniques**
Lauryn Bailey, et al.; SCIEX, SCIEX Concord, ON, Canada; Framingham, MA, USA

P-102   **Targeted Identification and Quantitation of Pesticide Residues using Advanced MRM Scheduling on a Triple Quadrupole LC-MS/MS**
André Schreiber and Lauryn Bailey, SCIEX, Framingham, MA, USA

P-103   **Application of High Resolution for Targeted Screening for Veterinary Drugs in Food**
André Schreiber, et al.; SCIEX, Concord, ON, Canada

P-104   **Data Processing for High Resolution LC-MS/MS for Target Quantitation and General Unknown Screening**
André Schreiber, et al.; SCIEX, Concord, ON, Canada

P-105   **Design of Experiment Versus "Change and Check": Method Optimization Strategies for the Determination of Microcystins as Markers of Algae Bloom Contamination in Surface Waters by LC-ESI-TOFMS**
Jason Weisenseel, et al.; PerkinElmer, Johns Creek, GA, USA

P-106   **Analysis of QuEChERS Extracts of a Variety of Foods for Pesticide Residues using Automated SPE Coupled to GC/MS/MS and LC/MS/MS**
Mark Hayward, et al.; ITSP Solutions Inc., Hartwell GA, USA

P-107   **A New Sorbent for Cleanup of Meat and Milk Extracts Prior to Multiresidue Veterinary Drug LC-MS Analysis**
Michael S. Young and Kim Tran, Waters Corporation, Milford, MA, USA

P-108   **Evaluation of A Modified Quechers Method for LC-MS Determination of Multiresidue Mycotoxins in Grain Flours**
Michael S. Young and Jeremy Shia, Waters Corporation, Milford, MA, USA

P-109   **Atmospheric Pressure Ionization Coupled to Tandem Quadrupole Mass Spectrometry for the Analysis of Pyrethroids in Water Samples**
Kenneth Rosnack, et al.; Waters Corporation, Milford, MA, USA

P-110   **Screening for Perfluoroalkyl Substances (PFASs) in Wildlife and Environmental Samples Using a Highly Sensitive LC-QTof MS**
Kenneth Rosnack, et al.; Waters Corporation, Milford, MA, USA

P-111   **Demonstration of Collision Cross Section Value Conservation Across LC and GC Analyses**
Jennifer A. Burgess, et al.; Waters Corporation, Milford, MA, USA

P-112   **A Novel Strategy to Screen and Profile Steviol Glycosides of Natural Sweeteners in Food Using Microfluidic UPLC Ion Mobility Mass Spectrometry**
Jennifer A. Burgess, et al.; Waters Corporation, Milford, MA, USA

P-113   **Determination of Triphenylmethane Dyes and Their Metabolites in Shrimp Using a modified QuEChERS Extraction and LCMSMS**
Gareth Cleland, et al.; Waters Corporation, Milford, MA, USA

P-114   **Facile Identification of Potential Pesticide Violations using Non Targeted Data Acquisition in Combination with an Integrated Scientific Information System**
Gareth Cleland, et al.; Waters Corporation, Milford, MA, USA

P-115   **A Single LC-MS/MS Method for Screening, Identification and Quantification of over 400 Pesticides in Complex Matrix without Compromising Data Quality**

Dimple Shah, et al.; Waters Corporation, Milford, MA, USA

P-116   **A Simple, Reliable and Fast LC-MS/MS Method For Determination and Quantification of Phthalates in Distilled Beverages**

Dimple Shah and Jennifer Burgess, Waters Corporation, Milford, MA, USA

P-117   **Determination of the Metabolites of Nitrofuran Antibiotics in a Range of Animal Tissues and Associated Products by Liquid Chromatography-Tandem Quadrupole Mass Spectrometry**

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

P-118   **Rapid, Direct Technique for the Discrimination of Meat Tissues Originating from Different Animal Species for Food Authenticity**

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

P-119   **Screening for Melamine, Cyanuric Acid and Dicyandiamine in Powdered Milk and Infant Formula using Liquid Chromatography- Mass Detection**

Eimear McCall, et al.; Waters Corporation, Wilmslow, UK

P-120   **A Rapid Screening Assay for the Simultaneous Detection of Antimicrobial Agents in Bovine Milk by Liquid Chromatography Coupled with an Accessible Mass Detector**

Eimear McCall, et al.; Waters Corporation, Wilmslow, UK

ORAL PRESENTATION ABSTRACTS

A-1   Streamlined Sample Preparation that Works So Well Needs a Catchy Name

Steven J. Lehotay

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Multiresidue pesticide analysis of foods has been conducted for more than 60 years, and the analytical methods have evolved greatly during that time. In 2003, Anastassiades et al. introduced the "quick, easy, cheap, effective, rugged, and safe" (QuEChERS) sample preparation method for multiclass, multiresidue analysis of pesticides in fruits and vegetables. QuEChERS takes advantage of the benefits of mass spectrometric detection to streamline sample preparation and cleanup to the minimum level that still provides acceptable quality of results without undue maintenance burden on the instruments. The QuEChERS approach was introduced at a time when a confluence of factors, including the introduction of QuEChERS itself, subsequently led to increased monitoring of pesticide residues in food worldwide. Although the chemistry of QuEChERS is very old and its details can be complex, its streamlined practical advantages and wide applicability make the QuEChERS template very elegant and flexible. We had to call the method something, and QuEChERS seemed appropriate because it lives up to its name. Currently, more than 20 vendors market QuEChERS products, and QuEChERS concepts are used in countless applications. At this time, the seminal publication by Anastassiades et al. (2003) has been cited >1400 times, and >1100 papers have been published using QuEChERS techniques in a myriad of applications. This presentation is aimed to provide a general overview of QuEChERS, including its background and some new developments.

A-2   Exploring the Limits of QuEChERS

Michelangelo Anastassiades

EU-Reference Laboratory for Pesticides requiring Single Residue Methods (EURL-SRM), hosted at CVUA Stuttgart, Schaflandstr. 3/2, 70736 Fellbach; Michelangelo.Anastassiades@cvuas.bwl.de

QuEChERS is nowadays worldwide the by far most widely used sample preparation approach in pesticide multiresidue analysis of food and feed. Since the publication of the original QuEChERS method in 2003 a vast number of QuEChERS-based applications have been developed covering not only the pesticide residues area but also many other fields such as contaminants, veterinary drugs and natural plant ingredients. The popularity of the QuEChERS approach is attributed to many aspects including its versatility, simplicity, low consumption of consumables, and the coverage of a broad range of GC- and LC-amenable compounds. Probably no other analytical method has been so extensively validated for such as broad range of compounds as QuEChERS. Still, the scope of pesticides that are amenable to QuEChERS is, limited, with certain highly polar, degradation prone or very volatile compounds requiring either modifications of the standard QuEChERS procedure or different analytical concepts such as the QuPPe concept for highly polar pesticides. This presentation will focus on such compounds.

O-1   Development and Evaluation of a Multi-Class Analysis of Nine Halogenated Environmental Contaminants in Salmon by Liquid Chromatography-Tandem Mass Spectrometry

Brittany J. Holmes

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In this study, a novel analytical approach for simultaneous extraction and quantification of five brominated and four chlorinated organophosphorus environmental contaminants in salmon was developed and evaluated using liquid chromatography triple quadrapole tandem mass spectrometry (LC-MS-MS). The method was based on a QuEChERS approach with acetonitrile extraction and clean-up with hexane and dispersive solid –phase extraction prior to LC-MS-MS analysis. The developed method was evaluated at 4 spiking levels and further validated by internal proficiency samples. Sample preparation for a batch of 16 samples took about 1 h/analyst, and LC-MS-MS analysis provided fast separation of multiple analytes within 13 minutes achieving high throughput. All but one analyte was recovered under the optimized extraction and LC conditions, of the remaining eight analytes six had recoveries between 70 and 120% and two had

recoveries at less than 20% with relative standard deviations less than 30% (n=9) for all but one analyte. The validated method was successfully applied for analysis of 649 salmon samples collected for a previous PDP survey.

O-2   Applying Metabolomics Technique to Reveal Matrices in the Pesticide Residue Analysis of Foods

Kuniyo Sugitate,[1] Machiko Saka,[2] Kazushi Mizukoshi,[3] Munetomo Nakamura,[3] Kazuyuki Yamashita,[1] and Sadao Nakamura;[1]

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[2]The Institute of Environmental Toxicology, Tokyo, Japan;[3] Japan Food Research Laboratory, Tokyo, Japan

Metabolites are the end products of the biosynthetic pathways in all living things. Many of them contain polar functional groups and can be analyzed using GC-MS with derivatization. We applied this technique to investigate what types of components are contained in the sample solution. First, different types of samples, i.e., potato, spinach, orange, soybean and brown rice, were analyzed according to the Japanese Positive List System (PLS) method. It was found that the common matrix components remained and that monoacylglycerols were largely responsible for the GC matrix effect. Based on these results, we could reduce the matrix effect using Z-Sep+ and Z-Sep/C18 (Sigma-Aldrich), and E-HyCu (Ehime University) which could remove not only the monoacylglycerols, but also fatty acids, flavonoids, sterols, etc. Next, the matrix components using the PLS method and the QuEChERS method were compared because of a rise of interest to the QuEChERS method. Sugars, flavonoids and fatty acids were not efficiently removed by the QuEcHERS method. Specific components in the samples, caffeine, capsaicins, gingerols and sesamin in the green tea, chili pepper, sesame and ginger, remained in the sample solutions after using any method. The probable reason for the remaining sugars and flavonoids was the shortage of water or insufficient dehydration. The reason for the remaining fatty acids was that the dispersive SPE was less effective than the packing SPE because PSA works as an ionic exchange. By incorporating the metabolomics technique, detailed matrix components were revealed, and this is a very helpful tool to develop the method.

O-3   Utilization of Multiple Walled Carbon Nanotubes (MWCNTs) in d-SPE cleanup and multi-Plug-Filtration-Cleanup (m-PFC) Method Development

Yuhong Qin[1], Nan Zou[1], Yanjie Li[1], Yongtao Han[1], Kejia Gu[1], Xuesheng Li [2] and Canping Pan [1],

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[2] Department of Agriculture Science, Guangxi University, P R China

In this study, MWCNTs were introduced into the cleanup procedure combined with QuEChERS method for pesticide/veterinary residues analysis in fruits, vegetables, teas, juice, meat, fish, milk, especially for difficulty-to-analysis samples. MWCNTs mixed with other sorbents were evaluated for their potential of adsorbing pesticide compounds or matrix interferences. Several types of pesticides, vertinary drugs were examined for these samples in method development and validation. Not only for LC-MS/MS or GC-MS/MS detection, IMS and Raman were also used to detect pesticides after using MWCNTs as d-SPE absorbents. The results showed MWCNTs had brilliant cleanup performance, even for difficult matrices like tea, leak or animal tissues. Furthermore, a novel rapid cleanup method based on MWCNTs in a packed column filtration procedure for analysis of pesticide residues was developed, which was carried out by applying streamlined procedure on multi-plug filtration cleanup (m-PFC) column with syringes. It is convenient and time-saving as it does not require any solvent evaporation, vortex, or centrifugation procedures. Sorbents were optimized for each matrix in d-SPE step, then they were adapted into multi-plug filtration cleanup columns. It's found that m-PFC could be used as an effective cleanup method. This method is expected to be widely applied for monitoring of pesticides at trace levels in the future for various agricultural commodities.

O-4   Development of a Molecular Imprinted Polymer SPE and LC/MS/MS Method for the Analysis of Pyridine Herbicides in Compost Samples

Michael J. Hastings, Ryan R. Brackman
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In the fall of 2012, Dow AgroSciences (DAS) and several Contract Research Organizations (CRO's) received compost samples produced by a commercial compost manufacturer which resulted in "auxin-like" plant injuries when used to enrich planting beds. Analysis of compost samples confirmed the presence of Aminopyralid and Clopyralid residues, but the levels reported varied significantly depending on the facility and the analytical method used. Differences in analytical

results from the CRO's have complicated 1) attempts to identify the original source(s) of the herbicide residues (i.e. which inputs in the compost stream contained the herbicide residues) and 2) subsequent label enforcement activities by state agriculture departments. During these product stewardship activities, it became clear that current analytical methodologies were unsuitable for the analysis of the low molecular weight, polar molecules in complex compost matrices at the 1ppb targeted detection level. This presentation will focus on the method development strategies employed to produce a simple, robust and easily-transferable multi-analyte method for the detection of pyridine herbicides in compost.

O-5   Addressing the Challenges in Marine and Freshwater Toxin Analysis

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The production of natural toxins by marine algae and freshwater cyanobacteria poses significant risks for human health, the seafood industry, drinking water supplies, and even for producers of certain dietary supplements. The complexity and range of these biotoxins means that reliable monitoring and regulation is a demanding task. Since the early dependence on animal bioassays significant developments have been made in methodologies for analysis of biotoxins, however, there are still challenges to be overcome. This talk will provide an overview on the issue and history of biotoxins, give insight on development of chemical analytical techniques, discuss efforts in the production of biotoxin reference materials, and will take a look at future directions in this interesting area of measurement science.

O-6   Detection of Microcystins: Does ADDA Add Up?

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Harmful Algal Blooms (HABs) have been recognized as a worldwide threat to human and ecological health, in both freshwater and marine systems. In August 2014 this threat reached national awareness when over 400,000 people in the city of Toledo, OH received a "do not drink" order, due to cyanotoxin breakthrough into finished drinking water. Toledo brought to light what scientists have known for years: we have inadequate means for quantifying cyanotoxins. As a consequence, regulators have an enormous challenge in setting safe drinking water standards, and providing standardized procedures for measuring the toxins.
We will use microcystin toxins to demonstrate these challenges. First we will discuss the most commonly applied drinking water threshold of 1 ppb, its origins and the role of interpretation in setting it. Then we will discuss the highly sensitive but poor specificity Enzyme-Linked Immunosorbent Assay (ELISA) that is the most widely used tool for measuring microcystins in water. The most popular ELISA is based upon the "ADDA" moiety, but as the title suggests, ADDA doesn't really add up for either toxin measurement, or for understanding human health risks. The ADDA moiety is also the basis for HPLC-PDA methods, and for a fluorescence-based method under development at Beagle, which both offer some improvements in specificity and in the case of the latter, sensitivity. We will conclude with a brief discussion of the "gold standard," LC-MS-MS, which is wrought with challenges related to poor sensitivity, high costs and limited availability of certified reference materials.

O-7   Management of Human Exposure to Marine Natural Toxins from Contaminated Seafood

Jonathan Deeds

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Contamination of seafood with natural toxins from the harvest area can cause consumer illness. Most of these toxins are produced by species of naturally occurring marine algae (phytoplankton). These toxins can accumulate in seafood when they feed on the algae or on fish or other primary vectors that have fed on the algae.   Most of the natural toxins that accumulate in seafood and cause illness occur in filter-feeding bivalve mollusks. To date, the FDA has established guidance levels for six toxin groups: Saxitoxins, responsible for Paralytic Shellfish Poisoning (PSP); Brevetoxins, responsible for Neurotoxic Shellfish Poisoning (NSP); Okadaic Acid and Dinophysistoxins, responsible for Diarrhetic Shellfish Poisoning (DSP); Domoic Acid; responsible for Amnesic Shellfish Poisoning (ASP); Azaspiracids, responsible

for Azaspiracid Shellfish Poisoning (AZP); and Ciguatoxins, responsible for Ciguatera Fish Poisoning (CFP). In the U.S., management of natural toxins in shellfish is achieved through the National Shellfish Sanitation Program (NSSP) through a combination of phytoplankton monitoring and toxin testing in harvest areas where these toxins have been shown to occur. Due to these programs, consumer illness from natural toxins in shellfish is rare in the U.S. The sporadic nature of toxin occurrence and an inability to routinely monitor harvesting areas for fish, makes the management of natural toxins in fish more challenging and reactionary in nature. There are also several toxins that occur naturally in certain species of fish (tetrodotoxin, gempylotoxin, etc.). These toxins are managed through import restrictions and/or the Hazard Analysis Critical Control Point (HACCP) regulation.

O-8   Harmful Effects of Marine Algal Toxins on Aquatic Wildlife

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In addition to threatening public health and damaging coastal economies, harmful algal blooms (HABs) and their toxins can harm fish and aquatic wildlife at various spatial and temporal scales. In particular, the marine algal toxins brevetoxins, saxitoxins, and domoic acid have been associated with multiple animal mortality and morbidity events. Brevetoxins produced by the dinoflagellate, *Karenia brevis*, cause widespread fish kills, are frequently associated with sick or dead marine mammals, sea turtles, and sea birds, and can disrupt benthic communities in the Gulf of Mexico. Saxitoxins, produced by several dinoflagellate species, cause mortalities of marine mammals, sea birds, and fish globally, and have caused several mass strandings of sea turtles in Pacific coastal waters of Mexico and Central America. Domoic acid, the only marine toxin of diatom origin, repeatedly causes acute and chronic illness and wide scale mortalities of sea lions and birds along the U.S. Pacific coast. Fish and aquatic wildlife can be exposed to marine biotoxins by direct contact with dissolved toxins or inhalation of aerosolized toxins. The most important route of exposure, however, is dietary, as concentrated doses of biotoxins can be present in food sources and can persist after blooms have ended. With the global expansion of HABs due to anthropogenic influences and changing climates, negative effects on aquatic wildlife are likely to increase. Information is needed on the effects of chronic sub-lethal biotoxin exposure and the synergistic effects of multiple biotoxins or biotoxins in combination with contaminants and other environmental stressors.

O-9   Screening of Phosphodiesterase Type 5 Inhibitors in Dietary Supplements Using Liquid Chromatography/Quadrupole–Orbital Ion Trap Mass Spectrometry

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Increased availability of dietary supplements in the market and popularity among consumers have been accompanied by increased adulteration of these products with active pharmaceutical ingredients. Phosphodiesterase type 5 (PDE5) inhibitors and their unapproved designer analogues represent an important group of adulterants that have been frequently used to develop or intensify the desired biological effect in sexual performance supplements. Considering that PDE5 inhibitors can interact with certain prescription drugs and limited knowledge is available on safety and efficacy of the designer analogues, the presence of such compounds in dietary supplements may represent a serious health risk to consumers. This presentation will discuss development and validation of a liquid chromatography–high resolution mass spectrometry (LC–HRMS) method for screening and identification of PDE5 inhibitor drugs and their analogues in various types of dietary supplements. The data acquisition approach on a Q-Exactive Plus instrument combined full-scan MS, data dependent MS/MS and all ion fragmentation experiments to obtain comprehensive information in a non-targeted fashion. We will describe development of exact-mass product ion spectra database for about seventy PDE5 inhibitors, provide criteria for reliable analyte identification, and discuss targeted and non-targeted screening for known and novel PDE5 inhibitors. Performance of the method in quantitative analysis mode will be also demonstrated.

O-10   Honey Adulteration: How to Detect the Unknown

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Consumer confidence on food authenticity is decreasing due to an increasing number of alert notifications related to food fraud. This is why there is a strong demand to detect fraudulent practices along the supply chain.

As a follow up of the horse meat scandal 2013 in Europe the European Commission published an EU action plan to tackle food fraud by listing the top 10 products that are most at risk of food fraud. In March 2015 the EU commission realizes this action plan by a recommendation on a coordinated control plan with a view to establishing the prevalence of fraudulent practices in the marketing of certain foods in March 2015. One of the target products is honey.

The detection and identification of foreign sugars added to honey is a real challange. It is known that there is no single method available which determines all possible foreign sugar sources. The untargeted approach is using techniques like isotope ratio mass spectrometry, spectroscopic screening, NMR and high resolution MS profiling. As a targeted approach LC, GC, GC-MS, LC-MS, ICP-MS and enzymatic tests allow the direct detection of marker molecules which are unique for foreign sugars but do not exist in natural honey. The presentation will give an overview of these methods and compare their advantages and disadvantages. All these methods have their limits and as a conclusion cannot detect all fraudulent practices on honey. Therefore a non-technical approach will be discussed as well by showing some opportunities to prevent food fraud.

O-11   Examination of the Adulteration, Counterfeiting and Contamination of Spices, Botanical Products, and Supplements by the detection of Heavy Metals and Potential Adulteration Compounds using ICP-OES and ICP-MS

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The consumption of botanical products has increased over the past two decades as consumers trend to what are perceived to be natural and high quality botanical products. The primary regions of spice and tea production around the have often been cited as having less stringent safety and quality standards in regards to consumer products. Products from these regions have been noted to contain a variety of adulterants and contaminants.

Common spices and botanicals in the US (Black Pepper, Red Pepper, Cinnamon, Mustard Seed, Cumin, and Turmeric) in various forms (i.e. spices, teas, condiments and supplements) were purchased at dollar stores, farmer's markets, chain stores, and online vitamin outlets. Products selected covered the range of preparations including organic products. Cryogenic grinding and microwave digestion were employed in sample processing. Physical and chemical screening methods were used to detect gross adulteration and counterfeiting. ICP-OES was used to determine the macroelement components (Si, Na, Mg, Fe, and K) that indicated possible adulteration or contamination. High percent levels of bulking agents including Silica and Sodium were often found in low cost spice and botanical samples indicating potential adulteration. ICP-MS was used to determine the presence and level of heavy metal contamination and adulteration. Most of the spice groups studied had many examples of high heavy metals content at the ppm level including very high lead levels which could indicative of adulteration by lead chromate or lead oxides.

O-12   New Approaches for the (Broad) Screening of Veterinary Drugs by Full Scan Accurate Mass Determination

Eric Verdon

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Mass spectrometry (MS) coupled with separation techniques like gas chromatography (GC) or liquid chromatography (LC) has become the superior analytical method of choice to control residues of veterinary medicinal products (VMPRs) in foods of animal origin. Due to their ability in separation, molecular mass identification and confirmatory quantification, these hyphenated techniques pave way for the detection of VMPRs in numerous biological matrices and food commodities such as milk, meat, fish, poultry, eggs, honey, feed, environmental samples. During the last decade the advances in chromatographic separation and in mass spectrometric techniques made easier the detection and quantification of chemical residues in biological matrices at ng/g level, especially with the LC-MS/MS technique. Today, new fairly-economically-viable bench-top mass spectrometers equipped with high resolution mass analyzers enable to measure the accurate mass of the detected substances and thus allow investigating the molecular structure of the compounds through determining their elemental composition. These developments have facilitated new expedients for control of veterinary chemical residues in biological matrices in the full scan mode. Meaning that these residual substances need no longer to be monitored on a targeted basis by using several methods aimed at testing specific molecules or families of molecules or even classes of molecules (such as antimicrobials, antiparasitics, … etc). This has opened arena for a novel strategy for chemical residue monitoring by using Full Scan mass analysis under High Resolution conditions and permitting

screening for all possible residual molecules in biological samples on post- targeted/non-targeted approaches. This concern will be displayed and illustrated throughout the presentation particularly focusing on the antimicrobial veterinary residue control.

O-13   LC-MS/MS Method for Determination and Quantification of Penicillin G and its Metabolites in Citrus Fruits Infected with Huanglongbing

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We have developed and validated an efficient method for the extraction, identification and quantification of penicillin G and its metabolite residues in a variety of citrus fruits (*e.g.,* lemon, orange, and grapefruit) using ultra high performance liquid chromatography coupled with triple quadrupole tandem mass spectrometry (UHPLC-MS/MS). By employing sequential liquid/liquid and solid-phase extraction techniques, we were able to remove essential oils and other substances from citrus matrices and extract penicillin G along with two of its metabolites (penilloic acid and penillic acid). The effect of matrix on the extraction efficiency was studied, and results indicate the applicability of this method to most of citrus fruits. Three fragment ion transitions per analyte were used for identification, which contributes to a high degree of selectivity. Penicillin G was quantified using a stable, isotopically-labeled internal standard benzyl (*i.e., d_5-*phenyl) penicillate, while penillic acid and penilloic acid were quantified versus standards in matrix for absolute recovery. We found typical recoveries of penicillin G around 90-100 % at fortification levels of 0.1, 0.25, 1, and 10 ng/g. Absolute recoveries for penillic and penilloic acids were found to be around 50-75 % depending on the matrix used. The limit of detection (LOD) of penicillin G and its metabolites was found to be 0.1 ng/g when 2 g of citrus was extracted. This method is useful in determining the human health risks associated with the persistence of penicillin G residues in citrus infected by Huanglongbing after antibiotic treatment.

O-14   Quantification of regulated and non- regulated lipophilic marine biotoxins by LC-MS/MS

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Consumption of shellfish (mussels, oysters, clams, etc) contaminated with biotoxins can cause severe intoxications in humans such as Diarrhetic Shellfish Poisoning (DSP). The complexity of the lipophilic marine biotoxins lies in the variety of physico- chemical properties such as carboxylic acids, sulfonic acids, amino and imino functionalities. In the European Union (EU) various toxin groups are regulated by legislation and these toxins should be monitored in official control programs. Since July 2011, the official EU reference method for control of lipophilic marine biotoxins in shellfish has been liquid chromatography- tandem quadrupole mass spectrometry (LC- MS/MS) based. The aims of this project were to produce a quick and robust method for the routine analysis and quantification of toxins. Whole fish tissues were analysed for many classes of regulated and non- regulated lipophilic marine biotoxins. A rapid method was developed, where satisfactory separation and peak shape were observed for the analytes within a five minute runtime under alkaline conditions. Single day validation showed excellent linearity for all toxins ($R^2 > 0.99$) over a wide concentration range (0.125 to 1.5 times the validation/ permitted level). Good mean recoveries (98 % to 102 %) and repeatability (RSD <12.5 %) were achieved for all regulated toxins.

O-15   False-positive and False-negative concerns with LC-QQQ in Production Lab: Risk Assessment

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What is the root cause of false-positive or false negative identification? When we talk about risks, are we on the same page? How could statistical risks be translated into regulatory and credibility risks? Will statistical risks of false-positive (Type I) and false-negative (Type II) have same risk factors associated with regulatory lab and private lab? We will attempt to depict a multi-scale overview and conduct risk analysis with technical tactics to address these risk concerns using Lean/Six Sigma tools.

O-16   Evaluation of emerging and unregulated drinking water contaminants and the impact of operations at American Water Facilities

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Current approaches to determine the presence of emerging (ECs) and other unregulated contaminants in the water bodies are mainly based on discrete measurements. Targeted chemical analysis has been traditionally employed to quantify the concentration of regulated and unregulated contaminants in the environment. The main disadvantage of this approach is to overlook the presence of unidentified chemicals in the water. Combined application of targeted chemical analysis by gas or liquid chromatography tandem mass spectrometry with modern analytical techniques based on advanced mass spectrometry, such as quadruple-time of flight, into environmental monitoring will provide insightful information to environmental specialists to determine the behaviour of ECs throughout water treatment. In this presentation an overview from different projects conducted by our research group to study the impact of operational conditions during full-scale drinking water and wastewater treatment on the fate of selected nitrosamines (unregulated disinfection by-products) and selected ECs utilized as performance and health base indicators (i.e. sucralose, estradiol, caffeine, triclosan, iopromide, DEET, gemfibrozil, atrazine) will be provided. Additionally, untargeted ECs were screened by LC/QTOF in MS^E mode in selected water samples at different treatment stages to determine the occurrence and fate of non-targeted ECs, including potential precursors for the formation of nitrosamines. The relationship between the concentrations determined in the water and changes in the plant operations and the usefulness of non-targeted analysis to identify unintended consequences of the operational changes will be also discussed.

O-17   Comparison of Analytical Methodologies for Analysis of Emerging Organic Contaminants in Water

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Emerging organic contaminants (EOCs) are ubiquitous and have been detected in surface and ground waters throughout the globe. These compounds are difficult to attenuate in conventional water treatment plants and have adverse effects to wildlife at environmentally relevant concentrations. Thus, the monitoring of these compounds is of great significance. When dealing with varying matrices and with the continual advancement of analytical equipment, it can be a struggle for even experienced analysts to choose the best form of sample clean-up and extraction step to get robust and sensitive data. Traditionally, labor intensive and time-consuming extraction techniques like SPE and LLE have been used for concentration of samples to achieve required detection limits. These methods require large amounts of sample (100-2000 ml) and solvents for extraction. Recently, the development of automated online SPE systems have given rise to the possibility of achieving similar detection limits with the use of just 1-10 ml of sample while significantly reducing cost, labor and time of analysis. With increase in sensitivity of modern mass spectrometers, large volume injection of the sample with no or minimal pretreatment has been mooted as a possibility for analysis of these contaminants. In this study, a single LC analytical method for the analysis of over 20 EOCs (ESI- and ESI+) using on-line SPE, were compared to direct large volume injection and offline-SPE followed by LC/MS/MS analysis. Factors like method reporting limits and ion suppression were considered. Further, the type of water matrix, cost and time of analysis will also be discussed for analysis of EOCs.

O-18   Non-target Analysis of E-Waste Samples from China using GC×GC-HRTOFMS

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This study aimed to chemically characterize new and emerging organic contaminants in environmental matrices surrounding an electronic waste (e-waste) site using comprehensive two-dimensional gas chromatography - high resolution time-of-flight mass spectrometry (GC×GC-HRT). The processing of e-waste in developing countries has become an environmental problem. Most electronics contain environmental contaminants such as brominated

flame retardants, which are a vector for environmental contamination. Though, potentially the most damaging environmental contaminants are produced by the improper processing of these components to form toxicants such as polybrominated dibenzo-*p*-dioxins and dibenzofurans. Chemical extracts were prepared from dust samples and particulate from an e-waste shredder. GC×GC-HRT provides unsurpassed analyte resolving power by leveraging four degrees of orthogonality. Peaks were separated in two orthogonal chromatographic dimensions and then deconvolved mass spectrometrically, with accurate mass as the fourth dimension. Peak True (Deconvoluted) Spectra were searched against commercially available library databases such as NIST 14 and Wiley 10 for tentative identification. Hits with a similarly greater then 800 (out of 1000) and a mass accuracy less than 1.5 ppm were considered to be correct in the absence of an authentic standard. Accurate mass was used to determine chemical formula for peaks with a library hit less than 800, and the mass spectrum was used to elucidate a structure where possible. Many legacy contaminants such as polychlorinated biphenyls, polycyclic aromatic hydrocarbons and chlorinated pesticides were identified, in addition to several other classes of chemical contaminants. This presentation will focus on the identification of new and emerging contaminants in e-waste related samples.

O-19   Antibiotic residue monitoring for animal food in Ho Chi Minh City, Vietnam

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A project named "Determine the Outbreak Mechanisms and Development of a Surveillance Model for Multi-Drug Resistant Bacteria" is now operating in Vietnam promoted by the Japan Science and Technology Agency (JST) and the Japan International Cooperation Agency (JICA). Vietnam is reported to present higher carriage rate of the drug-resistant bacteria compared to other countries. Under this project, a monitoring plan of residual antibiotics in animal food (beef, pork, chicken, egg, fish, shrimp) was conducted in Ho Chi Minh City from 2013. Samples were collected from slaughterhouses, wholesale markets and supermarkets. We used the simple method previously developed and monitored residual antibiotics such as sulfamides, quinolones and beta-lactams1,2. For egg or fishery products, extract steps were slightly changed. A part of samples were also tested by Premi®Test at sampling site. We analyzed over 600 samples from 2013 including beef, pork, chicken, fish, shrimp and egg. Four compounds were detected frequently, enrofloxacin and ciprofloxacin from fishery products and egg, sulfaclozine from chicken, tilmicosin from chicken and egg, and sulfamethazine from pork. Some of residues showed high concentrations (over 1 mg/kg). Overall, antibiotics were detected in about 15% of the samples collected in Ho Chi Minh City. Vietnamese government will tackle with the problems from just now on. And our project will contribute to such activities.

O-20   Recent Applications of High Resolution Mass Spectrometry in Veterinary Toxicology

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Veterinary analytical toxicology is a challenging field. In attempting to identify chemicals which may have caused health issues or death in an animal, the practitioner must have the ability to analyze for a vast array of toxins in matrices ranging from tissue to feed to environmental samples such as soil or water. For the sake of efficiency, it is desirable for individual analytical methods to encompass as many potential toxins and matrices as possible. While we're a long way from any technique which can handle all such chemicals, the use of full scan accurate mass LC-MS has allowed for methods which can cover a significant array of compounds in a single analysis with both high sensitivity and specificity. Our laboratory has found this technique valuable in handling a variety of analytical situations. Recent applications include the screening of commercial jerky treats for toxins and adulterants, analysis of passive water samplers for toxins relevant to wildlife, and analysis of hummingbird carcasses to assess potential exposure to pesticides. In this presentation, I'll discuss several of these applications with an emphasis on the value of high resolution accurate mass spectrometry.

O-21   Understanding Where the Specificity Afforded by "True" MS/MS is Fully Diminished Due to Increased Isolation Ranges Commonly Used in "Quasi-MS/MS" High Resolution Accurate Mass Measurements

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Fourier transform high resolution mass spectrometry (FT-HR-MS) requires long acquisition time, approximately 1 second, compared to commonly used low resolution and time of flight spectrometers. This is due to the fact that FT instruments measure ion package frequencies many thousands of times in order to achieve high resolution mass measurements. Unlike single stage FT-HR-MS, historically accepted MS/MS techniques typically have a Q1 isolation windows of about 1-3 Da which affords a high degree of specificity. For large screens narrow Q1 isolation windows require a very fast mass analyzer, typically capable of making a determination in less than 20 ms. One way to mitigate the problem of coupling parent isolation techniques with slow analyzers at high resolution is to widen the "parent" isolation window sufficiently, commonly about 50 Da, so that a range of m/z species are isolated and analyzed by MS/MS at high resolution. This approach allows the full range of m/z species of interest to be analyzed using a pseudo MS/MS technique.   As the isolation range is broadened so that high resolution mass measurements can be made, specificity based on the MS/MS character of the technique is diminished. The challenge lies in understanding at what point the specificity afforded by MS/MS is fully diminished due to increased isolation ranges and at what point is it useful to reduce resolution to afford narrower isolation windows. Our approach to the issue has been theoretically/mathematically as well as experimental.

O-22   Potential of GC Orbitrap MS Technology for the Analysis of Pesticide Residues in Food and Feed

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The analysis of pesticide residues in food and feed is a challenging task because of the high number (typically >800) of substances that need to be analysed in a diverse range of complex matrices. To cover the entire suite of pesticides, both LC and GC-based analyses are required. In both cases, a generic acquisition based on full scan MS detection is more straightforward than multiple reaction monitoring by triple quadrupole mass spectrometry. In order to obtain sufficient selectivity in the full scan mode, high resolution/high mass accuracy MS instruments are required. LC-based methods based on full mass range acquisition are gaining in popularity because of the capability to increase the scope of analysis in terms of the number of pesticides than can be detected, quantified and identified; and for the detection of unexpected (non-targeted) residues, in a single analysis. The advancement in hybrid HRAM MS/MS instruments means that these systems can, equal triple quadrupole performance.
The mass resolving power, mass accuracy, sensitivity, quantitative and qualitative screening performance of full scan high resolution MS coupled tor GC has lagged behind developments in LC, leaving an analytical capabilities gap. The results of the very first evaluation of different resolving powers, mass accuracy, scan speed, selectivity, sensitivity and linearity of a GC-EI-Orbitrap MS system for the analysis of pesticides in a variety of matrices will be presented. Initial results show that the system is capable of very low limits of detection, well below the maximum residue levels.

O-23   Determination of HR-MS Data Quality for Non-Targeted Screening of Food Matrices

Ann M. Knolhoff and Timothy R. Croley

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Many analytical screening methods for food samples target a compound or class of compounds. While advantageous, these methods will not detect other potentially harmful compounds that may be present. Non-targeted screening with liquid chromatography coupled to high-resolution mass spectrometry yields data with the necessary figures of merit for the generation of molecular formulae for compounds of interest. Specifically, the measured mass accuracy should be below 3 ppm and less than 5% isotope ratio deviation should be observed. However, data quality can be impacted by ion suppression, retention time changes, and coelution of compounds which could potentially limit the effectiveness of automated data analysis pipelines. Furthermore, food matrices are chemically complex and diverse and produce

complicated data sets. An Orbitrap and q-TOF were evaluated with a 48 compound analytical standard mixture spiked into different food matrices. Additionally, labeled analytical standards and their unlabeled counterparts were analyzed. Considerations for developing automated non-targeted data analysis workflows and factors that can contribute to impaired data quality will be discussed, such as chromatography, resolving power, and ion abundance.

O-24   Contribution of Sample Processing to Variability and Accuracy of Measured Residues

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Neither recovery tests nor proficiency tests provide information on the magnitude of variability and the bias of measured residues resulted from the inhomogeneity of processed sample material in terms of mass of test portion and the decomposition/evaporation of test substance during sample processing.   To demonstrate the potential consequences of sample processing, maize and lettuce were surface treated with a test mixture consisting of buprofezin, chlorothalonil chlorpyrifos, chlozolinate, cyprodinil, dichlorvos, etridiazol, fenhexamid, heptenofos, hexaconazole, imidacloprid, prochloraz, pyrimethanil, spiroxamine, tebuconazole, tecnazene, thiacloprid, and trifloxystrobin. Buprofezin and chlorpyrifos were used as stabile reference compounds. Treated materials were mixed with untreated ones in 1:9 ratio and then ground to fine powder (maize) at room temperature or chopped at room temperature and in the presence of dry ice (lettuce). The purified extracts were quantified with LC-MS/MS and GC-MS/MS. Grinding maize at room temperature resulted in loss of underlined substances ranging between 20 - 45% (dichlorvos) Only imidacloprid and thiacloprid showed no loss. In lettuce chlorothalonil and compounds shown in italics decomposed over 20% even in dry ice. The combined uncertainty of measurements expressed as relative standard deviation of buprofezin residues in 1 g test portion of maize increased by a factor of 2.5 compared to 10 g. For lettuce the increase of CV_{Sp} was 6 fold. The results highlight the importance of testing the performance of the applied procedures with samples containing incurred residues and performing recovery tests alone is not sufficient.

O-25   Theory of Sampling Guide to Quality Sample Processing

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Pierre Gy's Theory of Sampling identifies the types of heterogeneity in samples and their effect on analytical uncertainty. Gy's theory is just as relevant when processing samples within the laboratory to achieve a representative analytical sample. The 'sampling' conducted in the laboratory to reduce a bulk sample to a very small analytical sample can contribute more error than any other step in the analytical process. Particle size and mass of the analytical sample are critical components that should be carefully evaluated when developing sample processing methods to assure representative and repeatable analytical results. The sources of sample processing errors and the minimum analytical sample size will be discussed. Room temperature blending and cryo-processing techniques will be compared. Methods to evaluate the uncertainty in the sample processing step will be presented.

O-26   Cryogenic Milling: An Enabling Technology for High Throughput Residue Sample Preparation

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The effectiveness of the comminution approach used for bulk field samples limits the size of the subsample that must be extracted and analyzed to ensure an adequately representative and reproducible measurement. In many cases this subsample size restricts the residue method to the use of larger vessel formats, limiting downstream throughput. The introduction of a secondary fine milling step to this process using a subsample size already known to be representative can further improve sample homogeneity and allow direct method scaling to small high-throughput formats. The superior sample comminution achieved in cryogenic milling has been used in our labs to dramatically improve the overall throughput on many matrices (ranging from forages to undelinted cotton seed) across the range of our products including Glyphosate, Acetochlor, Dicamba. Metrics for homogeneity have been thoroughly characterized,

and have shown that sample homogeneity can be maintained down to sample sizes of 75 mg within our laboratory. This presentation will focus on performance characterization of secondary cryomilling along with the logistics of implementation of this technique for an efficient milling and cold dispensing workflow.

O-27   Assessment of Cryomilling Sample Processing of Plant and Animal Tissues for High Throughput Residue Analysis

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As the amount of sample used for residue analysis decreases due to increasingly sensitive analytical instrumentation and the incorporation of automation, the sample preparation step becomes ever more critical. In residue analysis, the initial sample size may vary from a few grams to several kilograms. In order to obtain accurate results, the processed sample must be sufficiently homogenous, such that a 100 mg aliquot will be representative of the initial bulk sample. This presentation will focus on results obtained from processing and analyzing both plant and animal tissues. The tissue samples were primary milled, followed by a secondary cryomilling step. Plant and animal tissue sub-aliquots of 100 mg were extracted and analyzed in a 96-well plate format, utilizing a liquid handler to automate the liquid transfers and a high throughput homogenizer to agitate the samples. The recoveries and % relative standard deviations obtained from the sub gram scale sample analysis were compared to method validation data generated using 1 to 5 g of plant or animal tissue sample.

O-28   Level of 2,4-D or Dicamba Residue found in Cucurbit Fruit from a Simulated Drift Scenario

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Cotton technologies with tolerance to glyphosate, glufosinate, and 2,4-D or dicamba will offer growers more flexibility in developing cotton weed management systems. However, adoption of these technologies will also increase the potential for damage to non-target specialty crops grown nearby. Vegetable production in Georgia has a farm gate value of nearly $1 billion including 33 vegetables produced for fresh market; 32 of which are sensitive to 2,4-D and dicamba. Thus, it is paramount that these vegetables be free of any illegal pesticide residues to protect the consumer as well as the grower. Cantaloupe and cucumber experiments were conducted during 2014; cantaloupe in the spring and cucumber in the fall at the Tifton Vegetable Park. Plots were 12 feet wide by 20 feet long with transplants planted into a 32-inch wide by 8-inch tall raised bed. Each bed was fumigated with 1,3-dichloropropene plus chloropicrin and Metam sodium and followed immediately with a low-density polyethylene mulch. Cucurbit production followed standard grower practices. Treatments were applied topically at 15 GPA with a backpack sprayer.
Cucurbits were treated with 2,4-D amine or Clarity (dicamba) at the 1/75X or 1/250X rate during three growth stages. The X rate for 2,4-D and Clarity was 1.0 and 0.5 lb ai/A, respectively. Herbicides were applied 54 (vegetative), 31 (bloom), and 18 (bloom/fruit) days before first harvest (DBH) of cantaloupe and 26 (vegetative), 16 (full bloom), and 7 (bloom/fruit) DBH of cucumber. A minimum of 0.7 inches of rain occurred between treatment and first harvest for both crops. Treatment separation of P = 0.05 was used except where noted differently.
Techniques utilized included QuEChERS extraction with analysis by LC-QTRAP. Residue levels detected by The Georgia Department of Agriculture were extremely consistent within treatments. For example, dicamba levels detected in cantaloupe at the 1/75X rate 18 DBH by replication were as follows: 0.01, 0.02, 0.015, and 0.011. For cucumber, dicamba levels detected at the 1/250X rate 7 DBH by replication were as follows: 0.004, 0.01, 0.01, and 0.005.

O-29   Quick Method for the Analysis of Select Midwestern United States Applied Pesticides in Surface Water from Agricultural run-off Using a Simple Sample Preparation Followed by UPLC/MS/MS Analysis

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The US EPA Chicago Regional Laboratory (CRL) developed a method for the qualitative and quantitative determination of select pesticides in the ppt to ppb concentrations. A quick sample preparation procedure using a few milliliter water sample was developed and is being utilized to determine pesticide concentrations in the creeks and streams from agricultural run-off. CRL also developed a method for the analysis of (Tri-n-butyl)-n-tetradecylphosphonium chloride

(TTPC), a biocide, in water and soil which has been used to characterize an industrial spill site.

One issue with the analysis of common chemicals is minimization or elimination of extraction or preparation steps that may contaminate a sample. The incorporation of the new state-of-the-art sensitive mass spectrometers allows quantification of these analytes at the ppt level with less sample manipulation. These procedures also save time, extraction and clean-up solvents. Many of the methods developed by CRL are incorporated into consensus standards such as the American Society for Testing and Materials (ASTM).

O-30   Development of LC-MS Based Multi-Mycotoxin Methods for U.S. FDA Compliance Testing and Surveillance

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Mycotoxins are toxic metabolites generated by fungi growing in foods. As a regulatory agency, the U.S. FDA needs reliable, efficient, and cost-effective analytical procedures to monitor the concentrations of mycotoxins in a variety of food commodities. Existing official methods used by the FDA laboratories target individual mycotoxins and require different procedures for confirmation. To improve efficiency, this study seeks to develop and validate methods that can screen, identify, and quantitate multiple mycotoxins in different food/feed matrices in a single sample preparation and analysis. Coupled with LC-MS (LC-MS/MS and LC-HRMS), different sample preparation procedures including immunoaffinity column clean-up, dilute-and-shoot, staple isotope dilutions and other available technologies haven been evaluated and optimized in food and feed matrices such as cereals, nuts, dried fruits, rice, wheat flour, milk, juices, and baby foods. The resulting multi-mycotoxin methods can identify and quantitate mycotoxins in different matrices using a single sample preparation procedure and LC-MS analysis, therefore improving the mycotoxin screening efficiency of the FDA laboratories in terms of identification, quantitation and sample throughput and enabling the U.S. FDA to more efficiently identify foods and feeds that may warrant regulatory actions.

O-31   FDA's Total Diet Study Program – An Update

Chris Sack

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The Total Diet Study (TDS) of the Food and Drug Administration (FDA) has been operating continuously since the late 1950's. In the TDS about 300 commonly eaten foods collected from multiple locations within the continental US are prepared for consumption, comminuted, and analyzed for selected nutrients, pesticides and other contaminants. The foods are chosen to characterize the US diet of children through adults, and cover a variety of matrices including raw agricultural commodities (as consumed), fast foods, recipe items, drinks, over 35 baby foods, and more. Four collections are analyzed each year. TDS residue data is used to calculate exposures to contaminants and assess risk for the US population.

Pesticide analysis of the TDS foods has advanced exponentially in recent years. Advances in technology to the LC-MS/MS and GC-MS/MS determination of targeted pesticides, and analysis by GC-MS in the full scan mode to screen for pesticides via spectral library, have increased the residues found by over 300 %. Implementation of these technologies has also allowed for simpler sample preparation procedures. A consolidated QuEChERS approach has been adopted for all TDS foods resulting in an expanded pesticide scope and increase in efficiency by as much as 900 %.

O-32   Matrix Effects within the Main Multirresidue Methods in Fruits and Vegetables

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The presence of matrix effects is one of the major concerns in food analysis. It affects seriously to the analyte signal and can lead to errors in the quantification, detection and ruggedness of the analytical methods. This work is focused on the evaluation of the co-extracted matrix components by the most common sample preparation methods for pesticide residues with the objective of predicting their potential negative effects.

Twenty five different commodities have been extracted by various methods by using LC-TOF-MS, mapping their natural compounds by retention time and accurate mass. Furthermore repeated analyses of spiked of the extracted samples with 120 pesticides were evaluated with all the extracted matrices. This way to show co-extracted matrix compounds allow seeing at a glance the complexity of the matrix and the interfering components distribution by polarity and molecular weight. The number, distribution and abundance of the natural components varied much between matrices includes in the same commodity group (i.e, high water content vegetables). "Difficult" providing a high number, concentration and distribution of natural components have associated a high suppression (i.e. orange and red onion >50 % of pesticides with high suppression) and problems with robustness systems. On the contrary, simplest matrices (i.e. apple, lettuce) do no present pesticides suppression higher than 30% and the consistency of the method is along a high number of injections. All the studied pesticides with high suppression >50% in orange matrix presented more than 75 co-eluting interfering species and Σsignal heigh > 2e7 counts. Pesticides free of suppression usually present less than 45 compounds and Σsignal heigh <1 2e7 counts. Dilution of the extracts was shown as an effective method to reduce the interfering compounds and diminishes the signal suppression for the majority of the pesticides in all commodities. The use of the statistical software Mass Profiler provided a powerful tool for evaluating and compares effectiveness of extraction methods through the co-extracted compounds.

O-33 Pesticide Data Program Sampling – Obtaining a Representative Sample

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One of the primary uses of PDP data is for dietary risk assessments. State-of-the-art analytical techniques are used to generate high quality data but the critical first step of sampling is easy to discount. In order for the data to be useable for making National inferences on exposure, a representative sample must be collected. In other words, reliable laboratory results begin with and depend directly upon the quality and timing of sample collection. PDP collaborates with Federal and State partners to ensure that sampling provides a statistically defensible representation of U.S. food supply so that PDP data reflect actual pesticide residue exposure from food. Key aspects include: designing a statistically-based sampling frame, covering all U.S. census regions, including all major fruit and vegetable production States, and collecting samples within hours of reaching consumers in order to represent pesticide degradation in storage and transit.

O-34 Plant and Animal Pesticide Method Radiovalidation Studies: Implications for Methodologies Used for Subsequent Food Safety Testing

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Plant and animal metabolism studies are required by EPA guideline 860.1300, Nature of the Residue - Plants, Livestock for pesticide registration. These studies determine the qualitative metabolic fate of an active ingredient when applied to crops or administered to livestock under simulated use conditions. Radiolabeled materials are employed so material balance can be monitored and the route of degradation can be determined. The position of radiolabel test in the molecule is critical to ensure the study accounts for all metabolic fragments. Based on the characterization and identification results as specified in the guideline, the total toxic residue (TTR) is established. Analytical methods are then developed to measure all components of the TTR which are used in subsequent residue trials. These residue trails provide the data upon which risk assessments are performed and are used to establish crop tolerances. Additionally a method is developed for tolerance enforcement. These tolerance enforcement methods must be radiovalidated by analyzing actual metabolism samples and showing concurrence with the metabolism study results. During radiovalidation, metabolism samples undergo the same extraction procedures proposed in the tolerance enforcement method. There is a need to demonstrate that the method accounts for ≥ 70% of the TTR in the aged samples, as identified in the metabolism study. Radiovalidation is often a misunderstood regulatory requirement. This presentation will discuss the importance of metabolism studies, leading to subsequent tolerance enforcement method development, radiovalidation, and the implications of these radiovalidated methods as they relate to pesticide screening for food safety.

O-35 **Food Safety and Security in the Modern World, Problems and Challenges Facing Scientists in Building Consumer Confidence in the Safety of our Food Supply. And, how do Nuclear Applications Fit into Food Security?**

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Our food supply needs to be Available, Affordable, Accessible, Nutritious and Safe. Compromising any one of these parameters affects global food security. Ensuring the safety and security of our food supply is significantly more complex today. The free flow of food products (including fresh and processed foods) across national borders poses a variety of challenges. Food safety scientists must be prepared for a range of situations that may arise adversely affecting the health and safety of consumers worldwide. Food safety and security involves ability to test not just for toxic chemical residues and microbial contamination but also for adulterants and contaminants and food authenticity, and traceability. Isotopic techniques significantly improve the analysis speed and reliability of food safety monitoring data and are essential tools in traceability studies. Challenges that we face today in ensuring global food safety and security along with involving the use of nuclear and complimentary techniques in food analysis will be discussed.

POSTER ABSTRACTS

P-1   Next Generation Matrix Removal Materials for Multi-Analyte Analysis

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Analysis of complex matrices often requires comprehensive sample preparation to extract analytes of interest at the appropriate concentration, while removing unwanted matrix co-extractives which can result in chromatographic interferences, ion suppression/enhancement in mass spectrometry, and accumulation in chromatographic flow paths. Developing concise and efficient sample preparation protocols for lipid rich matrices are of particular interest as current techniques include exhaustive cleanup steps or indiscriminately retain lipids and analytes of interest. This work demonstrates the benefits of using a new dispersive cleanup material with QuEChERS that dramatically reduces matrix co-extractives while maintaining acceptable quantitation accuracy and precision. Data will demonstrate the impact of superior cleanliness when conducting multi-residue analyte analysis in complex sample matrices using LC and GC. The ease of use, time and cost savings, minimal method development, and dramatically cleaner samples make this an attractive cleanup option for laboratories conducting trace analysis, especially in complex matrices.

P-2   Optimize Food Analysis with Miniaturized QuEChERS and an Ultra-Efficient Triple Quadrupole GC/MS

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There are many advantages in miniaturizing sample preparation. The use of less solvent reduces solvent cost and waste. Smaller sample sizes are easier to handle, store, and process in the lab. Reduction in sample amount provides cost savings on sample preparation sorbent and substantial decreases in cost associated with the use of labeled compounds as internal standards. With a smaller sample size, it may be possible to use additional labeled compounds for troublesome analytes that are cost prohibitive with larger sample sizes. We analyzed pesticides in food using miniaturized QuEChERS extraction and the ultra-efficient ionization source of the 7010 Triple Quadrupole GC/MS, which reduces injected sample volume by 75%. On average, limits of quantitation (LOQ) ≤ 10 ng/g were reached for 95% of the 126 pesticides studied in apple, carrot, and broccoli. Less matrix injected delivers prolonged uptime and sustained performance and, therefore, lower maintenance costs. By injecting only 25% of the standard 2 µL injection volume and implementing our recommended pesticide analysis method, we analyzed the pesticides at or below threshold MRLs of the EPA, EU, and Japan, 0.01 mg/kg (10 ng/g), which was adequate to monitor exposure.

P-3   Improving the Robustness of Daily Instrument Analysis through Enhanced Sample Matrix Removal

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Advanced modern analytical instrument detection systems such as LC/MS/MS, GC/MS/MS, GC/MS etc., have been used widely on trace level analysis of contaminant analysis in complex sample matrices. These instruments usually provide excellent detection sensitivity and selectivity, which allows the use of relative easy sample preparation techniques to extract analytes from sample matrix, such as QuEChERS, protein precipitation, dilute and shoot etc. However, these simple sample preparation techniques usually don't efficiently remove sample matrix interferences such as lipid and fatty ingredients, pigments. Therefore, the injection of these complex samples on instruments usually add more pressure on chromatographic columns and detection systems, resulting in more instrument downtime and frequent change of chromatographic columns. A new matrix removal material solves this dilemma by providing remarkable cleanliness for complex sample matrix, without additional steps, cost, or hardware. Data will be presented to demonstrate notable improvements on instrumental and chromatographic performance with cleaner sample matrices using the new material. The high performance and selectivity of this material make it an attractive option for those seeking to simple and easy sample preparations while maintaining acceptable instrument durability.

P-4   Screening for Hundreds of Pesticides in Fruits and Vegetables using a High Resolution Accurate Mass GC/Q-TOF with an Exact Mass Pesticide Library

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Most pesticide residue laboratories use GC/MS/MS and LC/MS/MS for their analyses because these tools are very sensitive and selective. However, these analytical techniques are for targeted analysis and they can only detect residues that are on the target list. In practice, this is usually a limited number of pesticides (typically 100 – 350 in total) because it becomes costly to purchase standards and to calibrate for hundreds of compounds. In addition, it can be time consuming to review the data. A screening method capable of looking for hundreds of pesticides without the need to purchase standards or calibrate would complement the targeted methods used by most labs. This talk describes a new pesticide screening approach using a high resolution accurate mass GC/Q-TOF with a new exact mass pesticide database/library. In principle, this method could screen for an unlimited number of analytes. The database/library contains entries for about 750 pesticides, making this an extremely comprehensive screening too. This approach was applied to spiked samples of fruit and vegetable extracts. Both methods identified more than 92% of the spiked pesticides at the 10 ng/mL level and more than 98% at the 100 ng/mL level.   Carbamates were the compounds most often missed, but this class of pesticides is normally done by LC/MS. If the difficult carbamates and a few other compounds, normally done by LC/MS, are eliminated from these data, the All Ions method was able to find 97% of the spiked compounds in the six matrices at 10 ng/mL.

P-5   Identification of Emerging Organic Contaminants in Water by Liquid Chromatography Time-of-Flight Mass Spectrometry

Jerry Zweigenbaum and Tarun Anumol

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Over 82,000 chemicals are approved for use in the US alone, with several hundred being introduced into the market daily. Thus, it is not a surprise that an increasing number of chemicals are being detected in our waters. Organic compounds like pharmaceuticals, endocrine disrupting compounds, industrial additives and pesticides collectively termed as trace organic compounds are drawing special attention because of their potential adverse effects to wildlife and humans at environmentally relevant conditions. The traditional analytical methodology for detection of these compounds has been by triple quadrupole instruments. However, this involves optimization of each individual compound on the instrument with synthesized analytical standards that can be very expensive and time-consuming. Economic feasibility requires the user to minimize the compound list being monitored to a smaller subset of 'indicator' compounds. However, with high resolution, accurate mass instruments like a time-of-flight mass spectrometer (Q-ToF-MS), rapid screening for thousands of compounds is now possible without the need for analytical standards and 'indicator' lists. In this study, different water qualities including treated drinking water, surface water and wastewater were evaluated using a liquid chromatograph coupled to a Q-ToF-MS for the presence of emerging organic contaminants. The samples were also compared to several databases including a pesticide and forensic toxicology libraries that contained several thousand compounds. This presentation will discuss the advantages and application of screening techniques with the LC-QToF-MS for environmental samples while providing an easy workflow that can be followed in labs performing high-throughput routine analysis for emerging organic contaminants.

P-6   LC/QTOF MS Determination of Pyrrolizidine Alkaloids in Dietary Supplements and Botanicals

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Pyrrolizidine alkaloids are known to be hepatotoxic and hepatocarcenogenic compounds and are found in common weeds that grow throughout Europe. Contamination of the plants that contain these secondary metabolites, such as Senecio jacobaea (ragwort), is common in botanicals used as dietary supplements and possess a health risk.   In this

work we show the LC/MS/MS determination of 25 pyrrolizidine alkaloids in both plants that produce them and dietary supplements. The accurate mass MS/MS of these compounds is presented and described. This characterization can be the bases of routine monitoring methods and determination of other toxic pyrrolizidine alkaloids in botanicals and dietary supplements.

P-7   Improvements in the QuEChERS Method for Multi-residue Analysis of Pesticides in Tobacco

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Tobacco is a crop that is impacted by a large variety of molds, insects, and viruses from seedbed to storage in warehouses after manufacturing. Growers and manufacturers will use various chemical agents, known as plant protective products, attempting to control these problems. Pesticides are commonly used on the tobacco leaf during the growing season. The impact of this manifests post-harvest and processing once the leaf is in its manufactured form. Pesticide residue is introduced through pyrolysis and is inhaled by the smoker and as an environmental tobacco smoke may be one source of pesticides in the body. Due to the use of more polar pesticides, LC-MS/MS technique has been implemented by laboratories. Improvements in the QuEChERS method allow for a quick and efficient sample preparation approach for the analysis of pesticides from tobacco with LC-MS/MS analysis. The calibration sets yielded correlation coefficient values (R^2) that were > 0.99 for 98% of the 131 pesticides spiked in tobacco. The average recoveries for the 131 pesticides fortified at 5 and 10 ng/g was 92%.

P-8   Determination of Imidazolinone Herbicides in Food of Plant Origin, By an Automated Extraction Platform

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Imidazolinone herbicides area family of five compounds includes: Imazapry, Imazapic, Imazethapyr, Imazamox, and Imazaquin. These herbicides are used to control a wide range of broadleaf weeds, by inhibiting the acetohydroxy acid synthesis (AHAS), which is the first common enzyme in the biosynthesis of branched chain amino acids.
The aim of this project is to evaluate the performance and versatility of the AutoMate-Q40 for the extraction of Imidazolinone herbicides. Liquid Chromatography coupled to a triple-quadrupole mass spectrometry (LC-MS/MS) was employed for the detection of these herbicides in agricultural commodities. Quantification was based on matrix-matched calibration curves with the use of internal standard to ensure method accuracy. By using the AutoMate-Q40 to streamline this extraction, it provides us with appropriate analytical results falling in the established method guidelines (range of 70-120% and RSD <20%) for the target compounds.

P-9   Tetracycline and Fluoroquinolone Residues in Meat, Chicken and Fish using Automation and New Consumables

Chris Shevlin, Zoe Grosser, **Horizon Technology** Salem, NH USA,
Sami Bayoudh and Kaynoush Naraghi, PolyIntell, Val-de-Reuil, France,
Curtis Hedman, Wisconsin State Laboratory of Hygiene, Madison, WI, USA,
Robert Buco, Richard Koeritz, and Zachary Lilla, Shimadzu Corporation, Marlborough, MA, USA

Tetracyclines are commonly used antimicrobial agents for agricultural livestock. With the concern that total veterinary use in the UK shows a ten-fold increase in use since 1969, government is moving towards legal action to allow the use of antibiotics for the treatment of health issues. Similar concerns are sparking discussions to change legislation in the USA, where 80% of antibiotic use is dedicated to livestock. Increased use of antibiotics in animal feed to enhance the growth of livestock has historically shown negative impacts on human health.
Fish farming is a growing commodity; however, the regulation of small and large fish farms will be critical for food safety on a global scale. According to the FAO, 37% of the fish produced is internationally traded. China leads the world in exports of aquaculture at 61%, while the USA leads the world in imports of fish, with over 50% sourced from aquaculture. Grown in close proximity, fish within an aquaculture environment can develop disease and impact aquaculture economic trade. The mechanisms for keeping fish healthy and free of disease is regulated within each country, often prompting the use of antibiotics in the water; however, the misuse of large antibiotic doses has increased attention on the impact to human health and thus the testing of aquaculture according to country specific food safety regulations.

Many food safety methods to test for agricultural contaminants in foods do not utilize current automation technology or new consumables, which can greatly reduce the impact of increased testing to ensure food safety and offer laboratories an efficient and timely testing workflow. With regulations increasing laboratory testing of samples, improving on these established methods to reduce the risk of backlog and human error is essential for timely reporting of results with confidence in the data. This paper will demonstrate the efficient methodology that can be developed for meat, chicken and fish, using automation and modern consumables.

P-10   Statistical Determination of Stability from Two Multi-Component Pesticide Mixes – Part Two

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A study verifying stability of pesticides, residues, and contaminants was based upon results from a study conducted in 2013. While the first study evaluated large mixes organized based upon instrument of analysis, this second study evaluated mixes based upon their presumed stability. These mixes were comprised of 374 presumed stable and 159 presumed unstable components. Additionally, components in both mixes were double the concentration from the previous study. Mixes were heat treated to simulate six month, one year, and two year time points. Randomized data obtained from the study was statistically evaluated by Analysis of Variance at the 95% confidence level. Results indicated that 386 compounds (seventy-three percent) across both mixes were stable through to the two year heat treatment time point, while 144 compounds (twenty-seven percent) across both mixes showed statistically relevant degradation between the control and six-month heat treatment. Interestingly, select compounds that were borderline stable in the first study exhibited lower stability when combined with the larger mix; likewise, a number of compounds with slight but statistically significant degradation from the first study demonstrated improved stability when contained in the smaller mix. This verification study supported the results observed with the 2013 study for the majority of the components, even with an increased concentration. Of notable importance was the observed change in stability of select components based upon the overall number of analytes in each mix. A compilation of both studies will be shown, illustrating the importance of conducting such studies when developing large component mixes.

P-11   Determination of Veterinary Drug Residues in Milk Using SPE and UHPLC-MS/MS with a Polyaromatic HPLC Column

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Veterinary drugs are widely used in food-producing animals. Milk is an important food commodity that is consumed by a large portion of the population, including infants. To ensure food safety and prevent the unnecessary exposure of consumers to veterinary drugs, it is important to test milk for drug residues. The use of multi-class, multi-residue (MMR) methods to detect veterinary drugs in milk is desirable. However, this can be challenging due to the large number of drugs with diverse physicochemical properties to be included in a method. Various extraction techniques were explored with the focus on suitably extracting a wide range of drug residues as well as achieving adequate limits of detection (LOD's). The final method utilizes a polymeric SPE extraction procedure, which was found to be the most suitable at achieving these criteria. UHPLC-MS/MS is the detection system of choice for veterinary drug residues analysis due to its high selectivity, sensitivity and ability to detect a wide range of compounds in a short run time. However, challenges still remain, including adequate retention and/or peak shape of polar drugs. In this work, LC separation is carried out with a polyaromatic stationary phase (Selectra® DA column), which exhibits alternative selectivity to a C18 phase and is capable of improved retention of polar drugs. This poster will outline a method for the determination of multiple classes of veterinary drug residues in milk using a polymeric SPE cartridge and UHPLC-MS/MS analysis using an aromatic stationary phase.

P-12   Analysis of Polychlorinated Terphenyls by GC-EI Triple Quadrupole Mass Spectrometry

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Polychlorinated terphenyls are chemically related to polychlorinated biphenyls and have similar chemical properties. They have very low electrical conductivity, high heat stability, and high resistance to alkalies and strong acids. They are non-flammable and insoluble in water. Use and marketing has been heavily restricted in the European Union in 1985. They have been marketed in the U.S. e.g. as Aroclor 5460, in Japan as Kanechlor C.

The analysis of this group of POPs consisting of three rings is performed less often compared to methods for polychlorinated biphenyls, which are frequently published and updated. The structural attribute increases the number of congeners, due to 14 theoretically substitutable hydrogen atoms. This leads to the need of a slower gradient in chromatography as the compounds elute quite closely. Furthermore, and one of the reasons for conducting this study, the analysis gets more prone to mass spectrometric interpretation and options as the steric conditions bear some interesting differences between compounds of the same sum-formula.

Method and instrument details

The contribution is going to describe the requirements and remarkable differences regarding the Mass Spec-experiments performed in details and the method itself can fulfill all regulations described in norms ISO 12766-3 or 38407-3.

P-13   Screening and Quantitation of About 200 Pesticides in Honey by an Integrated On-Line Extraction UHPLC-MS/MS System

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Solid Phase Extraction (SPE) is widely used for sample clean up before LC-MS/MS analysis. It is costly and time consuming. Here we present a simple, cost effective and sensitive procedure for screening and quantitation of pesticides in honey using an integrated On-Line Extraction (OLE)-UHPLC-MS/MS system for analysis of pesticides in honey.

A study using the EVOQ analyzed about 200 pesticides in honey using only one method with positive negative switching for about 400 MRM transitions. The measurements were conducted by dilute-and-shoot without sample enrichment. The honey was diluted 10-fold and filtered before injection. An YMC-Pack ODS-AQ, 10 μm, 10 mm x 2 mm (I.D.) column was used as trap column. An aqueous mobile phase was used to retain the pesticides on the trap column and to elute the monosaccharides in the honey out to the waste and then the valve switched to couple the trap column with analytical column for separation and detection. The preliminary spike recovery results showed that the full recovery achieved for most pesticides. The linear range was 1 to 1000 ng/g and the linear regression co-efficiency R^2 was >0.99.

P-14   Analysis of selected POPs with an Atmospheric Pressure Chemical Ionisation GC coupled to High-Resolution QTOFMS

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Trace analysis of polychlorinated dibenzodioxins (PCDD), polychlorinated dibenzofurans (PCDF) and polybrominated diphenyl ethers (PBDE) is one of the challenges in analytical chemistry. As they belong to the class of persistent organic pollutants (POPs) they are one of the major concerns in present environmental discussion. Due to the accumulation in the food chain it is of general interest to analyze them with good sensitivity and confidence.

GC-APCI coupled to a high resolution QTOF-MS offers a suitable and sensitive analytical tool for the analysis of those POPs. Here we report a method for Decabromodiphenyl ether (DecaBDE) and 2,3,7,8-Tetrachlordibenzodioxin (2,3,7,8-T$_4$CDD) using a Bruker 450-GC coupled to an impact II (Bruker Daltonik GmbH) as they are key substances for their compound classes. Quantitation was done in fullscan mode.

PBDEs are among the EU priority substances. DecaBDE is the most difficult PBDE to analyze, because it is less volatile and additionally thermolabile. DecaBDE showed a good response at a concentration of 1 pg on column, LOD was even lower. The analytical working range was between 1-40 pg on column.

2,3,7,8-T$_4$CDD is the most toxic substance of the PCDD/PCDF. 2,3,7,8-T$_4$CDD was detected as [M]$^+$ signal, LOD of 2,3,7,8-T$_4$CDD was <0.1 pg on column. The calibration curve showed an analytical working range between 0.1-2000 pg on column. Applying a higher collision energy caused a loss of COCl that could be used for confirmation.

Using GC-APCI coupled to high resolution QTOF-MS we achieved excellent detection limits at relevant environmental limit values.

P-15   **Multi-target Pesticide Screening using Atmospheric Pressure Chemical Ionization GC Coupled to High-Resolution Q-TOF-MS**

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The use of accurate mass QTOF-LC/MS with electrospray ionization for target pesticide screening enables the identification of hundreds of pesticides in a single run. On the other hand, GC/MS is well suited to these compounds and generally exhibits less matrix effects whilst producing lower chemical background. A pesticide standard mix consisting of a set of 60 representative pesticides was spiked into fruit and vegetable matrices selected according to their relevance in food analysis and according to their chemical characteristics as molecular mass, chemical composition, their polarity and volatility. The mix contained amongst others: Azinphos-Methyl, Chlorpropham, Diazinon, Dimethoate, EPN, Imazalil, Myclobutanil and Pirimicarb. 1 µl of each sample was injected and separated using a Restek Rxi-5ms capillary (30m, 0.25 mm ID, 0.25 µm film). The GC column was interfaced to an Q-TOF-MS (Impact II, Bruker Daltonics) with a GC-APCI source operated in both positive and negative ionization mode. Data were acquired from 50-1000 m/z at minimum of 4 Hz. All files were acquired with automatic mass calibration at the beginning of each GC/MS run with a perfluorinated calibration standard. All data were generated using automated mass calibration during each GC/MS run. This enables a mass accuracy < 3 ppm for all samples. The GC-APCI-Q-TOF-MS system was calibrated for quantification with the 60 pesticide standard in the concentration range of 0.05 to 500 pg/µl. Limits of quantification (LOQ) for most of the pesticides were found to be in the range well below 10 pg/µl with RSDs between 5 and 10 % (N=3).

P-16   **Development and Evaluation of a New Software Tool for Confident Identification of Pesticides in Food Extracts using High Resolution QTOF Mass Spectrometry**

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High-resolution time-of-flight mass spectrometry became an excellent tool in food safety. The accurate mass based inherent characteristics like sensitive wide scope screening together with retrospective and general unknown analysis capabilities make it an ideal tool for this work. We describe the development and evaluation of a new software tool allowing screening and quantification using ultra-high-resolution LC-QTOF accurate mass analysis. The new software allows applying the diagnostic ion concept, using enhanced confirmation criteria such as isotopes, adducts, fragments and qualifier ratios for reducing false positive detection rates. About 60 pesticides covering several compound classes were selected based on their relevance in food safety. Seven dilution series (0.01 ng/mL(ppb) – 5 µg/mL (ppm) based on solvent and five matrices (QuEChERS-extracts: cucumber, strawberry, wheat flour, leek, orange) were run in broad band CID (bbCID) mode: alternating acquisition of full scan and broad band CID spectra (2Hz). Automatic data evaluation is performed by the new software and a database with more than 700 pesticides including accurate mass, isotopic pattern, retention time, adducts and bbCID qualifier ions. Confidence in pesticide detection declines with decreasing compound concentration and increasing extract complexity due to the higher probability for interferences. Mass errors increase and isotopic pattern quality decreases, resulting in only tentative identifications. However, tentative results can be accepted as true positive findings even at low concentration levels, if the bbCID qualifier ions are detectable in the respective spectra. This allows the confident identification to significant lower levels compared to an approach using full scan data only.

P-17   **Automation of EPA Method 526: Determination of Selected Semi-Volatile Organic Compounds in Drinking Water by SPE and GC/MS.**

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EPA Method 526 is used to determine selected water-soluble, semi-volatile pesticides, herbicides, and additives in raw and finished drinking waters. One (1) liter water samples are passed through polystyrene-divinylbenzene solid phase extraction (SPE) cartridges and the analytes of interest are recovered with ethyl acetate and dichloromethane elutions of the dried sorbent bed. The samples are then concentrated and analyzed by gas chromatography/mass spectroscopy (GC/MS).

The PrepLinc™ Large-Volume Injection (LVi) system allows the user to pass large volumes of aqueous sample matrix through commercially available SPE cartridges and disks. The autosampler will accommodate 1 liter sample jars, therefore, the sample is taken directly from the container used to collect the water in the field. The analytes of interest can be eluted to collection tubes, or to an AccuVap module for concentration directly to autosampler vials. In this study, the labor-intensive steps of SPE conditioning, loading, elution and concentration were reduced to two steps: 1) loading samples on to the instrument and 2) programming the sequence with saved method parameters. This provided for a fast and simple, automated method for the labor intensive process of manually loading 1 liter of water to an SPE, subsequent elution, and concentration for analysis.

Baseline winter concentrations for the analytes of interest were established for 5 towns in rural, central Missouri. The identical sites were revisited in early May, following corn planting and typical application time for the analytes of interest, to establish drinking water affects in the monitored communities.

P-18   Organochlorine-Pesticides, Polychlorinated Biphenyls, and Polybrominated Diphenyl Ethers: An Automated Approach to Sample Preparation with Aquatic Biological Tissue Matrices.

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Organochlorine-pesticides (OCPs), polychlorinated biphenyls (PCBs) and polybrominated diphenyl ethers (PBDEs) are important, historical contaminates which are routinely monitored in environmental samples. These lipophilic compound classes are routinely found in biological tissues from aquatic environments owing to their persistence for accumulating in lipid, fats and oils, increased concentration up the food chain, and their low propensity for degradation in the environment. While many of these chemicals have been banned from manufacture and use in the United States since the late 1970s to the early 1980s, these characteristics of persistence and accumulation make them important priority organic pollutants to monitor in aquatic tissues for consumption advisories, endocrine disruption effects and general species health.

Due to the historic nature of these compound classes, methods for their extraction, isolation from co-extracted materials, and instrumental quantification are well established. However, most methods are time consuming and require manual handling at each step of the sample preparation process. Using the J2 Scientific PrepLinc™ system, lipid-rich aquatic tissue raw extracts were processed through automated, programmable methods to a final sample ready for gas chromatography-electron capture detection (GC/ECD) quantification. Gel permeation chromatography (GPC) followed by solid phase extraction (SPE) clean-up and fractionation resulted in two fractions for GC/ECD analysis with negligible manual manipulations. Fraction 1 (F1) contained PCBs and a few non-polar OCPs, and Fraction 2 (F2) contained the more polar OCPs and PBDEs.

P-20   GC-MS/MS TQ methods with short and long run times for limited- or extended-scope pesticide residue monitoring or import control purposes, using the new Dutch ("NL-") extraction method

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Official pesticide residue laboratories responsible for providing residue data for enforcement and the EU-coordinated monitoring program are facing increasing mandatory scope of analysis and shorter turnaround times in case of EU import control. LC- and GC-MS/MS triple-quad (TQ) are the state-of the-art "workhorse" techniques for routine analysis, showing excellent selectivity, sensitivity and ease of identification and quantification. These are used in combination with fast sample preparation and extraction methods. Scopes of the multiresidue methods can be adapted to the purpose of the analysis, in order to have short reporting deadlines with only a limited, priority scope or a more extended pesticides scope for monitoring purposes and complete consumer exposure calculations.

Two GC-MS/MS TQ methods with different chromatographic run times (22 and 35 min) with a scope of 171 and 310 pesticides, respectively, were developed on a Bruker Scion GC-MS/MS TQ instrument. GC- and MS-parameters were optimized in order to obtain the highest sensitivity and selectivity. The two methods were combined with the very fast and efficient Original and the New Dutch Mini-Luke extraction ("NL"-) method, without any necessary clean up. A homogeneous 15 g sample is extracted with 20 mL acetone (30 s via a Polytron homogenizer), followed by a partitioning step (30 sec) with 20 mL petroleum ether, 10 mL dichloromethane and 15 g Na_2SO_4, using the salting-out effect in order to assure good

recoveries for polar pesticides. The mixture is centrifuged (5 min at 3500 rpm), an aliquot (3 mL) of the extract is then evaporated to dryness (batchwise, on a water bath) and the residue is re-dissolved in 0.9 mL isooctane/toluene (9:1) and 5 μL is injected (via LVI-PTV) into the GC-MS/MS TQ.

The different methods were fully validated (EU DG SANCO criteria: average recoveries, 70-120%, RSD <20%), for 310 pesticides in lettuce and orange. Almost all analytes showed validated LOQs of 0.005 mg/kg. The estimated instrument LODs for the majority of analytes was well below 1 ng/mL for both matrices. The methods have been applied successfully in routine analysis of fruits and vegetables. Typical examples of monitoring and import control results will be shown. Also the equivalence validation of the original Dutch mini-Luke and the new NL-method will be shown.

P-21   Prediction of dietary possible metabolic residues in body by monitoring the formation of microbial metabolites of fungicide cyazofamid by Soil Fungus *Cunninghamella elegans*

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This study was performed to predict the possible metabolic residues in body by monitoring the formation of microbial metabolites from the sulfonamide fungicide, cyazofamid (4-chloro-2-cyano-N,N-dimethyl-5-*p*-tolylimidazole-1-sulfonamide) in soil fungus *Cunninghamella elegans* because they have the ability to metabolize a wide variety of xenobiotics, being similar to those in mammalian metabolism systems. The incubation of cyazofamid with *C. elegans* was carried out for 10 days and the metabolic reaction samples were analyzed using an Agilent HPLC 1100 series. Cyazofamid disappeared after 7 days of incubation, producing three metabolites. The metabolites were investigated for structure determination by Varian 500-MS^n mass spectrometer equipped with Agilent 1100 HPLC by ESI positive mode (needle voltage; 4000 V) from *m/z* 190 to *m/z* 350. Turbo Data Dependent Scanning (TurboDDS^{TM}, Varian) mode was used to obtain MS^3 spectra of ion *m/z* 341. Those three metabolites were identified as 4-chloro-5-(4-(hydroxymethyl)phenyl)-imidazole-2-carbonitrile (CHCN), 4-(4-chloro-2-cyanoimidazole-5-yl)benzoic acid (CCBA) and 4-chloro-2-cyano-5-(4-(hydroxymethyl)phenyl)N,N-dimethyl-1H-imidazole-1-sulfonamide (CCHS). Structure of CCHS was further unambiguously confirmed by 2D ^{1}H-^{13}C HSQC (heteronuclear single-quantum correlation) on NMR after its peak was isolated by fractionation/collection on HPLC with CAPCELL PAK C18 UG120 column. 2D ^{1}H-^{13}C HSQC NMR spectra were recorded on a 400 MHz NMR spectrometer in CDCl$_3$ at 292 K. As results for the possible metabolic residue formation in body, cyazofamid could be oxidized to CCHS at first step, then degraded to CHCN and further oxidized to CCBA. The metabolic system with *C. elegans* will be a powerful tool for predicting and identifying phase I metabolites that could be formed in mammalian systems.

P-22   Establishment of Fast Multiresidue Pesticide Analysis for Water Samples by LC-MS/MS and GC-MS/MS

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A rapid and simple multiresidue method base on simplified liquid-liquid extraction and liquid chromatography-triple quadrupole mass spectrometry (LC-MS/MS) or gas chromatography-iontrap mass spectrometry (GC-MS/MS) for simultaneous analysis of 139 (LC-MS/MS) or 108 (GC-MS/MS) pesticides in water was developed. For the sample preparation, the pesticide was extracted with acetonitrile before concentrated under nitrogen gentle stream. Detection on LC-MS/MS was performed by scheduled selected reaction monitoring (SRM) mode using positive or negative electrospray ionization, while detection on GC-MS/MS was performed by SRM mode for 2 groups of target pesticides. Quantitations were performed using matrix matched calibration curves at concentration ranging from 2 ng/mL to 200 ng/mL (LC-MS/MS) and 10 ng/mL to 1000 ng/mL (GC-MS/MS). Correlation coefficients (R^2) of calibration curves was >0.99 of almost target compounds. Method detection limits (MDL) were in the range of 0.04 ~ 0.56 ng/mL (fortified at 0.5 ng/mL or 1 ng/mL) for LC-MS/MS and 0.25 ~ 2.87 ng/mL (fortified at 5 ng/mL) for GC-MS/MS, respectively. To evaluate validity of the method, recovery tests were carried out with deionized water at spiking levels 1 μg/L, 5 μg/L (LC-MS/MS) and 5 μg/L, 25 μg/L (GC-MS/MS). For LC-MS/MS analysis, 66 ~ 71 % of pesticides satisfied the recovery criteria 70 ~ 120% (RSD ≤ 20%), while for GC-MS/MS analysis, 93 ~ 94% of pesticides satisfied. The established method was applied to underground water and paddy water samples from paddy field to detect 4 pesticides. As a result, the present method was proved to be efficient and rapid in multiresidue analysis of water samples.

P-23 **The Use of High Resolution Hybrid Mass Spectrometry and Liquid Chromatography for the Screening, Quantification and Identification of 451 Pesticides in Fruits and Vegetables**

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Due to the wide range of pesticides used on the production and postharvest storage of fruits and vegetables, it is vital for CFIA to develop methods that detect and unequivocally identify as many pesticides as possible. This poster presents key method development milestones using Ultra-high Performance Liquid Chromatography and Electrospray Ionization Quadrupole Orbitrap High Resolution Mass Spectrometry (UHPLC/ESI Q-Orbitrap MS) for the screening, quantification and identification of 451 pesticide residues in fruits and vegetables. A modified QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) method was used to extract pesticide residues from homogenized fruits and vegetables samples. Screening and quantification were achieved by operating the ESI Q-Orbitrap MS in full scan mode at 70,000 FWHM. The combination of high MS resolving power and UHPLC separation yielded 451 resolved compounds. The validation procedure consisted of apples, broccoli, banana, carrots, grapes, oranges, strawberries, lettuce, potatoes and tomatoes in triplicate, on two separate days for pesticides spiked at four concentration levels. The data was processed using TraceFinder 3.1 (Thermo) in "Quan" function, and exported to, and organized in Excel for SAS statistical analysis. Method performance met E.U. requirements and is reported elsewhere.

Identification was established by a combination of operating the ESI Q-Orbitrap MS in full scan/ddMS2 mode (data dependent) and processing data TraceFinder 3.1 in "Screening" function. A MS Library that includes fragments with exact mass, isotopic patterns, and retention times served as references to determine positive matches.

P-24 **Simultaneous Analysis of the Flonicamid and its metabolites TFNG and TFNA in Rice and Soybean using LC-MS/MS**

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The optimized method for simultaneous analysis of insecticide flonicamid and its two metabolites TFNG and TFNA in rice and soybean by LC-MS/MS was developed. In optimization of sample preparation, several methods were compared. When ethyl acetate was used for extraction, rice sample has good recoveries for all compounds but, soybean samples gave low recovery rate. Then a few different modified QuEChERS extraction were compared. The results revealed that the two metabolites were recovered under70% when PSA was used. Since the adsorption phenomenon by PSA was observed in two metabolites, the additional clean-up procedure with PSA was discarded. Also, acidified acetonitrile (containing 5% acetic acid) has increased the efficiency of extraction than normal acetonitrile. Final extracts were analyzed by SHIMADZU LCMS-8040™ triple quadrupole LC-MS/MS using electrospray ionization with selected reaction monitoring. To evaluate validity of the optimized method, recovery tests were carried out with untreated rice and soybean at spiking levels 0.02, 0.1 and 0.5 mg/kg. At all fortification levels, the accuracy and precision results satisfied 70-120% (RSD ≤20%) for all compounds. Quantitation was performed using matrix matched calibration curves at concentration ranging from 0.02 to 1.0 mg/kg. Correlation coefficients (R^2) of calibration curves was >0.99 for all target compounds. Limit of quantitation was 0.02 mg/kg. As a result, the optimized method was proved to be efficient and rapid in analysis of flonicamid and metabolites in rice and soybean.

P-25 **Use of ^{13}C isotope for Avoiding Linearity Problems in Time of Flight Mass Spectrometry**

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In the previous year's TOFs could not compete with triple quadrupoles in the field of quantitation because of low sensitivity and narrow linear dynamic range. The sensitivity problem has been reduced by introduction of modern ionisation sources and new designs of ion optics, but saturation effect linear range is still limited. This has negative influence on the quantitation.

A solution for the linearity problems can be quantitation by use of ions containing less abundant isotopes e.g. ^{13}C. Linearity range was investigated in QuEChERS extracts of tomato, cucumber, apple and orange. Extracts were spiked with 132 pesticides at concentrations from 0.005 to 1 mg/Kg. According to DG-SANCO guidelines [1] individual residuals cannot deviate more than 20%. This rule was used to determine the linear range. TOF quantitation of spiked samples was compared with the results obtained by triple quadrupole.

Among the investigated matrices the most challenging was apple. Using monoisotopic ions quantification up to 1 mg/Kg was possible in 29% of pesticides. Quantitation by use of ions containing one, two or three ^{13}C atoms increased the percentage up to 79%. In 15% of compounds other isotopes were applied (e.g. ^{34}S, ^{18}O etc.). In 6% of compounds quantitation was possible only up to 750 mg/Kg.

A comparison of TOF and QQQ 386 assays were done. Quantifying with monoisotopic ions 12.4% of results were in agreement (±20%) with results obtained by QQQ, whereas in case of quantification with isotopes 97.4%.

The described approach helps to resolve majority of quantitation problems present in TOF.

P-26   GC-(NCI)-QTOF Approach with Automated Accurate Mass Data Processing for Determination of 70 Pesticides in Vegetables

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Gas chromatography coupled to high resolution hybrid quadrupole time of flight mass spectrometry (GC-QTOF-MS), operating in negative chemical ionization (NCI) mode and combining single stage MS with MSMS experiment, has been explored for the automated accurate mass analysis of pesticides in fruit and vegetables. Seventy compounds were included in this approach.

Detection limits, recovery studies and repeatability were investigated at three concentration levels (1, 5, and 10 µg kg-1). A homemade database was developed and applied to an automatic accurate mass data processing. Mass accuracies of the generated ions were measured. When only one ion was obtained in the single stage MS, a new criterion for identification was proposed: to use the FS ion and a representative product ion from MSMS experiment. A total of thirty real samples were analyzed and 12 pesticides were detected.

A multiresidue acquisition and processing method has been validated for the simultaneous quantification of 70 pesticides in fruit and vegetables, providing very low LODs (75.7 % of pesticides had a LOD ≤1 µg kg-1), which is the main advantage of GC-QTOF operated in NCI due to its high sensitivity and selectivity.

The method was applied to analyze thirty samples from Almería, where chlorpyrifos, bupiramate and iprodione were the most commonly found pesticides with mass accuracies consistently below 5 ppm in at least one diagnostic ion.

From the results obtained, the combined use of HRMS and NCI promise to be a useful tool for analyze samples containing pesticides residues.

P-27   Multi-residue Determination of 69 Pesticides in Rice Straw Roughage by QuEChERS and HPLC-MS/MS

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Roughage is an important food source for feeding herbivores, which includes hay, straw, abortive shell and parts of the leaves, etc. Rice straw roughage is very vital for livestock in farming areas. However, the extensive use of pesticides in rice crops could cause unintended pesticides residues in abortive shell and straw roughages, and consequently influence the safety consumption of animal products. Until now, there has been no report on the pesticide multi-residue analysis in rice hull and straw roughage. The aim of the present work was to develop a multi-residue method for analyzing 69 pesticides in rice hull and straw roughage matrices. The extracting method and several different sorbents (C18, PSA and MWCNTs) for d-SPE cleanup were investigated and compared with respect to the recoveries and precision. For the

quantitative analysis, the recoveries ranged from 70-120% at the level of 1-100 µg/kg and RSDs＜29％（n＝5）were achieved for most of the target analytes (84% of the pesticides in rice straw and 80% of the pesticides in rice hulls). While the recoveries of some analytes were lower in both of matrixes due to their polar chemical structure. The LOQs for diverse pesticides ranged from 1-10 µg/kg. For the qualitative analysis, the retention times and the ion ratios of different matrix-matched standard solutions kept consistent with the reagent-only standards.

P-28   Utilization of Multiple Walled Carbon Nanotubes (MWCNTs) in d-SPE Cleanup and Multi-Plug-Filtration-Cleanup (m-PFC) Method Development

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In this study, MWCNTs were introduced into the cleanup procedure combined with QuEChERS method for pesticide/veterinary residues analysis in fruits, vegetables, teas, juice, meat, fish, milk, especially for difficulty-to-analysis samples. MWCNTs mixed with other sorbents were evaluated for their potential of adsorbing pesticide compounds or matrix interferences. Several types of pesticides, vertinary drugs were examined for these samples in method development and validation. Not only for LC-MS/MS or GC-MS/MS detection, IMS and Raman were also used to detect pesticides after using MWCNTs as d-SPE absorbents. The results showed MWCNTs had brilliant cleanup performance, even for difficult matrices like tea, leak or animal tissues. Furthermore, a novel rapid cleanup method based on MWCNTs in a packed column filtration procedure for analysis of pesticide residues was developed, which was carried out by applying streamlined procedure on multi-plug filtration cleanup (m-PFC) column with syringes. It is convenient and time-saving as it does not require any solvent evaporation, vortex, or centrifugation procedures. Sorbents were optimized for each matrix in d-SPE step, then they were adapted into multi-plug filtration cleanup columns. It's found that m-PFC could be used as an effective cleanup method. This method is expected to be widely applied for monitoring of pesticides at trace levels in the future for various agricultural commodities.

P-29   Antibiotic Residue Monitoring for Meat and Egg in Ho Chi Minh City, Vietnam in 2014 - 2015

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Using of antibiotics in livestock and poultry production will lead to antibiotic residues in food. Antibiotic residues in food will influence the long-term health of consumers. In this study we have analyzed over 200 samples including pork, poultry and egg. The samples were collected at super market and whole sale market in Ho Chi Minh City (HCMC) from 2014 to 2015. Residual antibiotics including sulfonamides, quinolones, β–Lactams in meat samples were analyzed by LC-MS/MS technique we previously developed[1]. Meat samples were also tested by Premi®Test at sampling site. The preparation method applied to egg samples for LC-MS/MS analyses was developed newly. Sulfadimidine was frequently detected from pork. Some of pork samples showed high concentrations (about 1 mg/g) and these were matched to the results of Premi®Test. The quinolone group including enrofloxacin, norfloxacin and ciprofloxacin, was often detected in egg samples. A few egg samples contained very high level of enrofloxacin which were around ten times the MRL in Vietnam. Tilmicosin antibiotic was also detected in some egg samples. The result shows that antibiotic still using in Vietnam for livestock and poultry production. Vietnamese authority needs to establish antibiotic monitoring system in food especially for meat, poultry and egg.

P-30   Monitoring of Illegal Compounds, including Anti-impotence Drugs, Anti-obesity Drugs and their Analogues in Foods

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Illegal compounds have been constantly detected in foods including dietary supplements, which are meant for sexual enhancement or weight loss. These illegal compounds are mainly sildenafil, tadalafil, sibutramine and their analogues. The adulteration of foods with these drug analogues may threaten public health because their safety profiles have not been characterized. The aim of this study was to monitor of adulterants in foods. We collected 44 foods advertised as sexual enhancers or slimming products in the Korean market and on the Internet from January to April in 2015. We analyzed 73 compounds such as anti-impotence drug, anti-obesity drugs and their analogues using high performance liquid chromatography/photodiode array detector (HPLC/PDA) and LC- mass spectrometry /mass spectrometry (LC-MS/MS). In this study, we identified illegal compounds in 13 of 44 samples. The levels of detected compounds were as follows; sildenafil 0.5-170.8 mg/g, tadalafil 17.5-83.1 mg/g and chloropretadalafil 0.3-0.8 mg/g.

P-31   Method for Determination of Pahs in Liver Samples of Green Sea Turtles (*Chelonia mydas*) Using HPLC-FLD

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Determination of chemical residues in complex matrixes is always a challenge due to low recoveries and matrixes effects present during analysis. The scientific literature reports many methods for biological matrixes such as liver samples, however the consume of long periods of time for the extraction step, usually more than 10 hours, and the use of high quantity of organic solvents like in the soxhlet extraction method, makes its application unfeasible.In this way, a new method has been adjusted mixing ultrasound-assisted solid liquid extraction (USLE) and a QuEChERS clean-up step for determination of 15 EU priority PHAs residues (napththalene, acenaphthene, fluorene phenanthrene, anthracene, fluoranthene, pyrene, benz[*a*]anthracene, chrysene, benzo[*b*]fluoranthene, benzo[*k*]fluoranthene, benzo[*a*]pyrene, dibenz[a,h]anthracene, benzo[g,h,i]perylene, indeno[1,2,3-c,d]pyrene) in liver samples of green sea turtles. The method involves extraction of 0.3 g of dried liver sample using 20 mL of hexan:dichloromethane = 1:1 (v/v) assisted by ultrasound extraction during 15 min (twice) and clean-up step using 150 mg $MgSO_4$ and 50 mg of PSA and finally 20 μL injected into the HPLC-FLD for the respective analysis. The method achieved LQs from 0.1 to 1.7 ng g^{-1} and showed no matrix effect. For evaluate the recovery performance two levels were tested (8 and 200 ng g^{-1}), recoveries from 71.9 to 102.2 % with CV ≤ 12.5% and 75.3 to 93.4 % were achieved respectively and the uncertainty of the method was from 3 to 24 %. The method showed to be adequate for the determination of 15 PAHs residues in green sea turtle's liver samples.

P-32   Potential Role of PAHS in Green Sea Turtles Fibropapillomatosis

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It has been known that fibropapillomatosis (FP), a disease characterized by development of benign skin tumors, poses a significant threat to green sea turtles' conservation. The polycyclic aromatic hydrocarbons (PAHs) may be co-factors in the pathogenesis of these disease due to their carcinogenic and genotoxic potential. The aim of this work was to evaluate the possible relations between 15 PAHs (napththalene, acenaphthene, fluorene phenanthrene, anthracene, fluoranthene, pyrene, benz[*a*]anthracene, chrysene, benzo[*b*]fluoranthene, benzo[*k*]fluoranthene, benzo[*a*]pyrene, dibenz[a,h]anthracene, benzo[g,h,i]perylene, indeno[1,2,3-c,d]pyrene) and (FP) in 44 liver samples of green sea turtles with and without FP. The specimens were captured at three Brazilian feeding areas, according to monitoring of Projeto TAMAR-ICMBio and Projeto Biopesca: Praia Grande and Ubatuba (São Paulo State) and Vitória (Espírito Santo State). For evaluate the severity of FP, the size and the number of tumors were considered. Results ofPAHs were analyzed in samples from specimens without FP (n=20) and with three FP levels: low (n=13), medium (n=4) and severe (n=7). Mean ∑PAHs were 4.4, 15.4, 11.2 and 21.7 ng.g^{-1} (without, low, medium and severe groups respectively). The statistical analysis

for 44 animals displayed a positive correlation (r=0.61) for ∑PAHs Vs the presence of FP with a significance of ≥ 95%. In addition, the Tukey´s test displayed that two groups (severe and medium) had significant difference in comparison with low and without FP, on the other hand, there were no statistical differences between "low" and "without FP"groups. Even though we found significant concentrations of PHAs in the liver samples of green sea turtles that could suggest a potential role in FP development, more studies are necessary to fully understand and corroborate if these pollutants are really involved in the onset of FP.

P-33 **Monitoring of PAHs in Liver Samples OF Green Sea Turtles (*Chelonia mydas*) With and Without Fibropapillomatosis Captured at Three Brazilian Feeding Areas**

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Due the constant contamination of the marine environment by organic and inorganic compounds by human activities, the presence of the organic contaminants like pesticides, polychlorinated biphenyls (PCBs) and polycyclic aromatic hydrocarbons (PAHs) have been reported by several researchers worldwide, and its impact on marine animal populations is not fully understood. The association between occurrence of some compounds in the tissues of marine animals and a greater prevalence of infections or physiological dysfunctions has been signaled by many studies. There is the suspect of an involvement among pollution and development of neoplasia in some animals, such as the fibropapillomatosis (FP), disease considered a relevant threat for marine turtles, which are included in the Red Lists of threatened species of IUCN and Brazilian fauna. By these reason, 44 liver samples of green sea turtles with and without FP were analyzed. The specimens were captured at three Brazilian feeding areas, according to monitoring of Projeto TAMAR-ICMBio and Projeto Biopesca: Praia Grande and Ubatuba (São Paulo State) and Vitória (Espírito Santo State). A total of 15 EU priority PHAs residues (napththalene, acenaphthene, fluorene phenanthrene, anthracene, fluoranthene, pyrene, benz[*a*]anthracene, chrysene, benzo[*b*]fluoranthene, benzo[*k*]fluoranthene, benzo[*a*]pyrene, dibenz[a,h]anthracene, benzo[g,h,i]perylene, indeno[1,2,3-c,d]pyrene) were monitored. The extraction method were carried out using adjusted mixing ultrasound-assisted solid liquid extraction (USLE) and QuEChERS clean-up step, and the analyzes were performed by HPLC-FLD. The analyzes of 20 liver samples from green sea turtles without FP showed a maximum concentration of 25.1 ng g^{-1} (dw) of ∑PAHs, on the other hand, 24 samples (green sea turtles with FP) showed a maximum concentration of 30.8 ng g^{-1} (dw) of ∑PAHs. The results confirmed the presence of these compounds in liver samples of the green sea turtles with and without FP examined, all of them captured at feeding areas of Southeast of Brazil.

P-34 **Determination of Organochlorine Pesticides in Organic Quinoa Grains**

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There is currently great interest in environmental pollutants in relation to food security; Previous studies show that POPs could be found in small amounts, even in organic food, for these reasons in this study 12 organochlorine pesticides residues (p, p'-DDD; p, p'-DDE, or , p'-DDD, or, p'DDE, Dicofol, α - endosulfan, β - endosulfan, endosulfan sulfate α - BHC, β - HCH, heptachlor and mirex) were determined in quinoa grains from organic production systems, sampled in the Region of Puno – Peru; the QuEChERS method was used for pesticides extraction and gas phase chromatography coupled to an electron capture detector (GC-μECD) for the detection and quantification of the pesticides. The method was validated using a quinoa sample without pesticide residues used as a blank and spiked to 0.001 and 0.10 mg kg^{-1}; mean recoveries for pesticides ranged from 73.66 to 128.09% with RSD ≤ 16%; the method showed linearity of r2 ≥ 0.99 and the Limits of Quantification (LQs) ranged from 0.001 to 0.010 mg kg^{-1}. The results indicate that there is no matrix effect and reveal the presence of three organochlorine pesticides (p, p'-DDD, p, p'-DDE and mixex) in five different samples; two of them showed concentrations of p, p'-DDE above 0.01 mg kg^{-1}. The results demonstrate the susceptibility of quinoa grain to the accumulation of toxic substances and show the need to establish new criteria for organic certification.

P-35 Antibiotic Residue Monitoring for Freshwater Products in Ho Chi Minh City and Thai Binh, Vietnam

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Vietnam is now one of the most rapidly developing countries and one of the richest in freshwater products. Last year, we presented the monitoring results of antibiotics in food collected from Ho Chi Minh City (HCMC) and Nha Trang local markets. To reveal the antibiotic residue in freshwater products, the monitoring was continued, and overall 294 samples have been analyzed. Samples were collected from eight local markets and one whole sale market in HCMC and three local markets in Thai Binh (TB) from August 2013 to March 2015. In TB, freshwater products were harvested from households and streams. Thirty-nine compounds including 14 sulfonamides, 12 quinolones, and 7 β-lactams were analyzed. The number of detected samples was 27 of 176 analyzed samples in HCMC. In contrast, only one of 118 analyzed samples was detected in TB. Enrofloxacin and its metabolite ciprofloxacin were most often detected, and the detection rates in HCMC were 13.2% and 4.0%, respectively. Sulfamethazine, which is the major antibiotic detected in pork and chicken, was also detected. Sulfamethoxazole and trimethoprim were also detected. Enrofloxacin has been included in the list of chemicals and antibiotics prohibited for use in fishery production and trade in Vietnam, but it was shown that the percentage of freshwater samples that contain residual enrofloxacin is relatively high. Our results imply that the inappropriate use of antibiotics in aquaculture, and/or contamination due to human activities still persists.

P-36 Risk Based Analysis of Chemical Hazards

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The Food Safety Modernization Act (FSMA) signed into law by President Obama on January 4, 2011. The law enables the US Food and Drug Administration (USUSFDA) to better protect public health by focusing on preventing food safety problems rather than reacting to issues after they occur. Many HACCP programs focus on microbial hazards. Chemical hazards were relegated to identifying sanitizing agents, mechanical grease, machinery lubricants and facility pest control products. However, FSMA specifically identifies and calls out pesticides, drug residues and unapproved food and color additives as reasonably foreseeable hazards to consider. Chemical contaminants can cause serious damage to your customer's health and have a major effect on your business. While rare, food products contaminated with, for instance, an unregistered pesticide is more common and will trigger, in the United States, a Class II recall. It is necessary to protect your product from chemical contaminants and identify the hazards in your ingredients before they can affect your final product. It is often not realistic to test every lot of ingredient for every chemical contaminant and one approach is to develop a monitoring program for your most risky ingredients. The initial challenge to developing an effective monitoring program is identifying your high risk ingredients. A risk analysis model is employed to identify and weigh risk for ingredients. The risk analysis process follows a structured approach with three distinct but closely linked components: 1.)Risk assessment 2.)Risk management 3.) Risk communication.

P-37 Analysis of 16 Antibiotics in Feedstuffs Using Ion Trap LC-MSⁿ

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Multi-analyte methods are a beneficial means for reducing analysis time and reagent consumption. LC/MSⁿ is well suited for analyzing multiple analytes in a short time and identifying analytes in complex matrices, such as animal feeds. An LC/MSⁿ screening method is being developed and validated for sixteen antibiotics in swine feed, dried distiller's grain and soybean meal using a linear ion trap mass spectrometer. The antibiotics are from six different antibiotic classes:

amphenicols, fluoroquinolones, sulfonamides, ionophore coccidiostats, streptogramins and macrolides. For feed sample analysis related to trace levels of concern or zero tolerance, identification of antibiotics at less than 1 ppm may be necessary. For label claim verification, the level of antibiotics may be tens to hundreds of ppm. The multi-antibiotic method being validated is capable of identifying all sixteen antibiotics at less than 100 ppb, as well as at a 40 ppm spike level on the three matrices. Precision (%RSD) results for all sixteen antibiotics fall within 0.9 – 23% for all spiking levels tested, from less than 0.10 ppm to 40 ppm for swine feed and dried distiller's grains. Results for fourteen of the sixteen antibiotics have recoveries of 68 – 117% for all spiking levels for both of these matrices. Analysis of proficiency test and incurred feed samples for Tylosin, Monensin, Sulfamethazine and Sulfathiazole showed 86 – 130% agreement, compared to either AAFCO HPLC or spectrophotometric methods for the antibiotics.

P-38 Developing an In-house Proficiency Test Sample for Aroclor 1254/1260 Mixture in Fish to Meet the ISO/IEC 17025:2005 Requirements

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The state of Minnesota issues consumption advisories for fish caught in the state based on the levels of polychlorinated biphenyls (PCBs) and mercury. Due to the site-specific background the PCB levels are quantified as Aroclor 1254/1260 mixture. The authors developed an analytical method for the Aroclor mixture in fish using GCMSMS and sought ISO/IEC 17025:2005 accreditation for the method. ISO/IEC 17025:2005 Standard states *participation in interlaboratory comparison or proficiency-testing programs* as part of quality control procedure that monitors the validity of a test. There was no external PT provider who could assure that the Aroclor mixture would be in their PT. The authors' laboratory worked on creating an in-house PT sample for the method with the help from three other laboratories: California Animal Health & Food Safety, Connecticut Agricultural Experiment Station, and Mississippi State Chemical Laboratory. Five large fish from a Mississippi river site contaminated with the Aroclor mixture were captured. Each fish was filleted and homogenized. Twenty aliquots of each homogenized fish (PT candidate sample) were analyzed by the authors' laboratory. Five (5) aliquots of each PT candidate sample were analyzed by the three laboratories using the analytical method they have been using to analyze their routine samples for PCBs. Therefore, the PT candidate samples were analyzed under the following different measurement conditions: dates, locations, operators, and measuring systems. Statistical analyses were conducted on the data collected by the 4 laboratories to select the best PT sample candidate.

P-39 The Use of Liquid Chromatography/High Resolution Mass Spectrometry in the Analysis of Pesticide Residues on Produce

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Analysis of pesticide residues in produce using liquid chromatography/mass spectrometry is typically performed by targeting specific transitions from parent ions to product ions to provide a sensitive signal with minimal noise. High resolution mass spectrometers however, allows for sensitive signals with low noise without targeting specific transitions. These instruments instead operate in full scan modes, specificity is provided by looking at the exact molecular weight of targeted compounds. Confirmation of the pesticide identity can be provided by all ion fragmentation. All compounds are analyzed using the same scan functions and all the data is collected. These instruments can therefore be used to determine hundreds of different pesticides in a single analytical run without individually tuning and setting up for each pesticide. Furthermore, the possible presence of a non-targeted compound could be probed long after the sample was actually analyzed. This presentation will discuss the use of this technique within the State of Connecticut's Market Basket Survey of pesticide residues in fruits and vegetables. Produce samples are prepared with a QuEChERS based extraction, and analyzed on an Agilent 1200 liquid chromatograph interfaced to a Thermo Exactive high resolution mass spectometer The method allows for quantitation of pesticide residues at the parts per billion level.

P-40 Photodegradation of 2,6-dichloro-4-nitroaniline (DCNA) in Freshwater and Saltwater

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The salinity of seawater can influence both the overall rate of degradation of chemicals and impact the distribution

and types of photoproducts. The photodegradation of the fungicide 2,6-dichloro-4-nitroaniline (DCNA) was measured in both distilled water and simulated seawater to determine the degree of differences of the degradation rate and product distribution. Solutions of DCNA at a concentration of 1 ppm were prepared and irradiated for 24 hours in an Atlas SUNTEST XXL+ photochamber that mimics the wavelength distribution and intensity of sunlight. Samples were withdrawn at 2, 4, 6, 12, and 24 hours for analysis and analyzed for residual DCNA using an Agilent 1260 Infinity High Performance Liquid Chromatograph. Products including the small aliphatic acids succinic acid, maleic acid, fumeric acid, and oxalic acid as well as nitrate, nitrite, bromide, and chloride were measured using a Thermo Dionex ICS-5000+ ion chromatograph. The half-life of DCNA was calculated to be 7.6 hours and 8.2 hours respectively in distilled and simulated seawater. Preliminary analysis of degradation products including chloride and nitrate suggested DCNA is degraded via a photonuclephilic substitution processes. Further analysis is needed to determine if differences in salinity impact the distribution of observed products.

P-41   Pesticide Sediment Partitioning and Exposure Modeling

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Current regulatory exposure modeling scenarios assume pesticides will evenly distribute through the top 5 cm of sediment after entering an aquatic system, however it is possible this overestimates the mass loading of chemical to sediment. Freshwater pond tests have been designed using Savillex Teflon tubes partially filled with soil and light water flow in order to simulate partitioning behavior under freshwater pond or estuarine conditions. Sediment extractions are currently performed using a Buchi Speed Extractor and chemical residues are measured using an Agilent 1260 Infinity HPLC system. Testing has shown recoveries ranging from 39.61% - 50.21%. Methods are still being developed in order to maximize recovery before continuing with fresh water pond simulations. Extraction recoveries are being refined for 2,4-D, quinclorac, bentazon, fipronil, flutolanil, bifenthrin and cypermethrin, which encompass a wide range of solubility and sediment sorption physical properties. The completion of this project will allow for better predictions of pesticide exposure for benthic communities.

P-42   Water Solubility Measurements of Atrazine and Fipronil, in Freshwater and Seawater

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Salinity has been reported to impact the water solubility of organic chemicals entering marine ecosystems however there surprisingly is scarce data available on the universe of chemicals potentially entering seawater. Impacts on solubility would corresponding impact on chemical sorption as well as overall bioavailability and thus impact overall exposure estimates. The water solubility of fipronil and atrazine were measured in fresh water (pH = 5), 0.01 M phosphate buffer (1.22 g/L, pH = 7), and sea water (salinity 32 g/L, pH = 8) at 25˚C using an equilibration shake-flask method. The water solubility of fipronil was 1.9 mg/L in seawater, 5.8 mg/L in buffer and 4.8 mg/L in distilled water, which were similar to the value of 1.9 mg/L reported in the literature. The water solubility of atrazine was 50 mg/L in seawater, 65 mg/L in buffer, and 55 mg/L in distilled water, which were slightly above the reported value of 34.7 mg/L at pH = 7 (22˚C) using EPA shake-flask method. While values varied slightly from literature values, possible due to small variations in experimental conditions such as temperature, the overall data still indicated overall reductions in solubility in seawater vs distilled water.

P-43   A High-Throughput and Sensitive Method For Quantitation And Identification Of Chloramphenicol in Foods of Animal Origin Using UHPLC-MS/MS

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Chloramphenicol (CAP) is a broad-spectrum antibiotic and potential carcinogen that is banned by WHO and also banned in the United States, Canada, European and most Asian countries in food products. However, due to its broad activity

and low cost, the illegal use still remains. In this study, a fast, high-throughput and sensitive UHPLC-MS/MS method for quantification and identification of CAP was developed and validated to determine sub-μg/kg levels of CAP in infant formula, honey and seafood (shrimp, fish meal, oyster and mussel) samples. The sample preparation consists of dispersing the sample in water followed by extraction with acetonitrile, partitioning the aqueous and organic layers by the addition of sodium chloride, exchanging a portion of the acetonitrile layer into water and then analysis by UHPLC-MS/MS using negative electro spray ionization mode. The isotopically labeled internal standard d5-CAP is added to compensate for matrix effects. The calibration range was from 0.025 to 5.0 ng/mL. The average recoveries in infant formula, honey and seafood fortified at the EU Minimum Required Performance Limit (MRPL) of 0.3 μg/kg ranged from 89.9% to 111% with relative standard deviations from 1.0% to 5.6%. Method criteria for data acceptance and an approach for handling complex samples with severe ion suppression were established.

P-44 Screening and Identification of Adulterants in Weight Loss Supplements by UHPLC and High-Resolution Accurate-Mass Detection

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The dietary supplement market segment has been growing worldwide at 5% or more yearly for the last several years. Weight-loss products belong to a very popular supplement category. Unfortunately, those products are being frequently adulterated with synthetic weight-loss drugs with anorectic or laxative effects (such as sibutramine and its analogs) and also with antidepressants to suppress side-effects of these drugs. Therefore, screening and identification of a wide range of adulterants are prerequisites for many supplement distributing companies to ensure consumer safety, comply with regulations and protect their brand. We developed a high-resolution/accurate-mass screening and identification method using a Q-Exactive Plus instrument, which provides a wide analytical range allowing for detection of both very low (contamination) levels and also very high (adulteration) levels, which can occur in real-world samples. A combination of full scan MS-data dependent MS/MS and all ion fragmentation (AIF) was employed to acquire data for both known (targeted) and unknown (non-targeted) compounds. Our data processing workflow incorporated an in-lab generated database of potential weight-loss supplement adulterants to match analyte retention time, precursor mass, isotopic pattern and up to ten exact-mass fragments. Additionally, the AIF option allowed for retrospective analysis of the data and search for compounds not included in the database. A simple dilute-and-shoot sample preparation was used and evaluated in the analysis of a wide range of weight-loss supplement sample types (capsules, tablets, tinctures, oils, liquids, powders, and gummies) that we purchased and analyzed together with over-spiked extract portions to establish sample preparation and instrument detection/identification performance.

P-45 Highly Polar Pesticide Analysis in Food Samples by LC- MS/MS

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Food safety laboratories involved in pesticide monitoring typically employ multi-residue LC-MS/MS methods for the quantification of large numbers of pesticides. However, several highly polar pesticides are extremely challenging to analyse and therefore it is necessary to utilise single-residue methods, including derivatisation for some compounds. The application of single residue methods requires considerable laboratory resources relative to the number of compounds analysed; hence very few laboratories target these compounds on a regular basis. The goal of this project was to develop a fast, sensitive and simple methodology for a range of challenging highly polar pesticides that require single-residue methods, by as few multi-residue LC-MS/MS runs as possible and without the need for derivatisation.
Mobile phase screening was performed on a range of different analytical columns (Obselic R, SM-C18, Zic-HILIC, Zic-cHILIC, Hypercarb, Phenyl hexyl) capable of various interactions (reversed phase, normal phase, HILIC and ion exchange). Signal to noise was compared using ESI +/- and APCI +/- to select the most appropriate ionisation technique. ESI+/- was found to be superior for nearly all compounds. The most suitable columns for the target compounds were determined to be the ZIC-HILIC and the Hypercarb as they permitted the use of multi-residue methods. Ionisation source parameters were optimised for each method. The final methods were evaluated using apple matrix with each compound in the concentration range of 0.005 – 0.2 mg/kg. The developed multi-residue methodology provided significant time savings in comparison to previously utilised single residue methods.

P-46   Direct Determination of Trace Hormones in Drinking Water by Large Volume Injection at Sub ng/L Levels Using LC-MS/MS

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Endocrine disrupting compounds interfere with the body's endocrine system and produce adverse developmental, reproductive, neurological, and immune effects. Consequently several hormones are routinely monitored by the US EPA in drinking water as part of the Unregulated Contaminant Monitoring Rule program (UCMR3). This program demands extremely challenging sub ng/L reporting levels that are typically achieved using time consuming SPE, as in EPA method 539. In this study a fast, selective and highly sensitive method has been developed using a direct high volume injection cycle with a fully optimised LC/MS/MS. This method reaches the required reporting levels without the need for extensive sample preparation using conventional SPE methods.
The target hormones in this study included: estrone, estriol, 17-β-estradiol, equilin, androstenedione, testosterone and 17-α-ethynylestradiol. Ammonium fluoride as an aqueous mobile phase additive was found to significantly improve response for all studied hormones in comparison to ammonium hydroxide. Using the developed method on the LCMS-8050 detection limits ranged from 0.0058 ng/L for testosterone to 0.33 ng/L for 17-α-Ethynylestradiol. Linearity was assessed from 0.5 times the required reporting level to 100 times the reporting level. All seven hormones achieved excellent correlation coefficients greater than $R^2 > 0.999$, using external standards for quantitation, a linear fit and a 1/C weight. Peak area reproducibility (n=8) was assessed at the reporting level and a high concentration. At the low concentration repeatability was < 4.3 %RSD, with the exception of 17-α-Ethynylestradiol (12.2 %RSD). At the high concentration repeatability was < 3.9 %RSD for all compounds.

P-47   Measurement of Antifungal Residues in a Treated Cannabis Crop

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Prevention of fungal infection is an essential aspect of cannabis production. Although proper growing and harvesting practices should be followed to prevent spoilage, in some cases antifungal agents have been applied to crops suffering from severe infection. Residues from these agents pose a risk to consumers and testing by a sensitive and selective technique such as LC-MS-MS is needed to determine whether a specimen is contaminated. In the present work, samples from a cannabis crop treated with the antifungal myclobutanil were prepared with a QuEChERS-style extraction and measured by liquid chromatography-triple quadrupole mass spectrometry.

Representative samples were collected 2 and 7 weeks after application to determine how much remained over time. Myclobutanil was detected at both time points, however in the specimen collected 7 weeks after application the level had dropped almost 20-fold, from a very high level of 192 ppm to 10.6 ppm at the latter time point. The LC-MS-MS method also screened for over 200 pesticides, however other than myclobutanil, no other pesticide residues were detected. All plant samples were provided by a licensed grower and samples were analyzed in a licensed laboratory in full compliance with Washington state law. The antifungal was applied to the plants by a licensed applicator.

P-48   Analysis of Veterinary Drug Residues in Livestock and Fishery Products

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Residues of veterinary drugs in the food supply pose a risk to consumer safety. A wide variety of different compounds may be present including growth promoters, antibiotics, and other drugs to promote animal health. Rapid and accurate detection of trace quantities of these substances requires highly sensitive and selective techniques such as LC-MS-MS. Four different animal tissues (shrimp, chicken, pork, and salmon) were tested for the presence of 89 veterinary drug residues. The drug residues represent a wide variety of compounds including antibiotics, antifungals, steroids, growth

promoters, and others. Samples were prepared with a QuEChERS-style extraction and analysis using a UHPLC-triple quadrupole mass spectrometer.

Performance for each analyte including detection limit and recovery was determined using appropriate replicates and spiking studies. For nearly all compounds, recoveries were between 70% and 120%. Instrument robustness was determined by endurance testing and RSDs lower than 5% were found over several hundred consecutive samples. This sensitive and selective method for determination of veterinary drug residues in animal products provides rapid and accurate measurement of a broad variety of potentially harmful substances.

P-49   Pesticide Screening in the Non-Regulated Medical Marijuana Industry by GC-MS/MS

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Since medical marijuana (MM) was legalized in California in 1996, 23 states and Washington, D.C. have passed laws allowing its use for a variety of medical conditions. From a consumer safety point-of-view, quantitation of the pesticide residues in MM products has begun to attract wide interest. There are several problems associated with analysis of pesticide residues in MM. First and foremost, there are very few regulatory guidelines established to define which pesticides to include or what the detection limits should be, and secondly the matrix is very complex with significant interferences. Finally, sample load is growing exponentially, so the chosen method must be quick and easy to perform. Trace level pesticide analysis in complex food matrices have been done for many years with similar challenges, thus many of the analytical protocols emerging for the MM matrix are based on these well-established techniques. Triple-quadrupole GC-MS/MS operated in MRM mode provides significant sensitivity and selectivity, but method development can be expensive and time consuming. This poster describes streamlined method development process for analysis of pesticide residues in MM using a QuEChERS sample preparation method, followed by GC-MS/MS detection and quantitation.

P-50   Pesticides Analysis by On-line SFE-SFC-MS/MS for Improved Sample Preparation, Analysis Time, and Sensitivity, Plus a Wider Range of Analyte Polarities Measured

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The goal of this poster is to address four areas of improvement for pesticide analysis over traditional methods using on-line Supercritical Fluid Extraction – Supercritical Fluid Chromatography – Mass Spectrometry/Mass Spectrometry (SFE-SFC-MS/MS). Those areas are reduced sample preparation time, faster separation of analytes, increased sensitivity, and analysis of a wider range of analyte polarities. Sample preparation can be reduced from a 35 minute QuEChERS method to a 5 minute method with the patented on-line SFE-SFC interface. Polar compounds typically analyzed by LC-MS/MS can be analyzed faster by SFC-MS/MS. Sensitivities can be improved over LC-MS/MS methods with the new multi-patented splitless backpressure regulator (BPR). In addition, SFE-SFC-MS/MS enables analysis of a wider range of compounds from non-polar to polar than currently available by GC-MS/MS or LC-MS/MS. The whole process can be fully automated by adding a 48 sample rack changer on the front end.

P-51   A Simple and Rapid Extraction Method for Chlorinated Pesticides in Poultry Meat Using Solid Phase Extraction and GC/ECD

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Pesticides contamination is not limited to fresh produce. Poultry meat used for food consumption is also exposed to contaminants at levels that can pose harm to the human population. The extraction of pesticides from poultry meat presents challenges that includes and not limited to time and solvent consumption when conventional liquid-liquid extraction techniques are adopted. This food matrix itself is complex due to the presence of proteins and lipid. Non-selective extraction methods do not eliminate all interferences and eventually results in decreased column lifetime and increased system maintenance. Presented is a Solid Phase Extraction (SPE) method, developed to selectively extract chlorinated pesticides from poultry meat using Strata® Alumina-N solid phase extraction cartridges. The extraction is simple and rapid and utilizes minimum solvents. Following the extraction, the GC analysis is performed using Zebron® ZB-MultiResidue™-1 column and Electron Capture Detector (ECD). The ZB-Multiresidue-1 is a proprietary stationary phase with Engineered Self Cross-linking™ (ESC) that offers selectivity necessary to separate chlorinated pesticides that are structurally similar. The optimized GC method results in a 15.0 min total runtime for all the chlorinated pesticides, eluting all analytes within 11 min. The SPE GC/ECD method for pesticides analysis from poultry meat outperformed the traditional procedure with results of decreased laboratory space requirements, reduction of hazardous waste and significant reduction of labor consumption, leading to greater laboratory productivity. From an analytical perspective, the method presents cleaner chromatograms that are free from matrix impurities and suitable for quantitative analysis.

P-52   High Resolution Accurate Mass (HRAM) Collision Energy Profile of Residues of Concern for Food Safety

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High resolution accurate mass (HRAM) fragmentation patterns for hundreds of pesticides, toxins, and drugs have been determined using at least ten different collision energies from 10 to 105 eV using a Thermo Scientific Exactive Orbitrap. The fragmentation patterns of representative compounds are displayed. The fragmentation patterns provide useful information for multi-residue method development. These can be especially useful for co-eluting compounds where one compound may contribute an interfering ion for the quantitation of another. In the development of multicomponent residue mixes for the use in rapid LC/MS/MS source optimization solutions, having all parent and fragment ions be unique by unit mass resolution at all energies is essential.

P-53   Cleanup of QuEChERS Extracts using SBSE for LC/MS/MS Determination of Pesticides in Food Products

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One of the most important aspects of reducing pesticide exposure is monitoring of pesticide residues in foods. A number of analytical methods have been developed, many of them based on traditional liquid-liquid extraction in combination with GC-MS or LC-MS. The QuEChERS (quick, easy, cheap, effective, rugged, and safe) sample preparation methods have been developed to help monitor pesticides in a range of food samples. The dispersive Solid Phase Extraction (SPE) used to clean up these extracts can leave co-extractants which can result in interferences such as ion suppression with the analytical results.
Stir bar sorptive extraction (SBSE) is a sorptive extraction technique based on polydimethylsiloxane (PDMS) coated stir bars. SBSE was developed to concentrate nonpolar analytes from aqueous solutions, and has recently been shown to effectively extract and concentrate PAHs from QuEChERS extracts while eliminating matrix interference for GC/MS analysis.
In this study we describe the potential benefits of using SBSE to concentrate pesticides from QuEChERS extracts and provide additional clean-up from matrix interferences during LC/MS/MS. Pesticides concentrated on the SBSE phase were recovered by liquid desorption providing better analytical sensitivity for the pesticides being monitored with reduced matrix interference. Manual steps such as evaporation, reconstitution, and dilution as well as the subsequent LC/MS/MS analysis of the final extracts can be automated to improve laboratory productivity for monitoring pesticide residues in foods.

P-54   Streamlined Sample Preparation Methodology to enable Higher Recovery, and minimize loss of Pesticides, Fungicides and Antibiotics by LC/MS or GC/MS.

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The most critical aspects of reliable food contamination analysis are the reduction of interferences from the sample matrix and analyte recovery. Traditionally, SPE, SLE, Liquid-Liquid, syringe filtration, and centrifugation have been used to reduce matrix interference prior to LC/MS analysis. However, these techniques are time consuming, adversely impact recovery, require expensive consumables, and use large amounts of solvent. Improved sample prep methods were developed using eXtreme|FV or the eXtractor|3D for contaminant analysis were conducted in milk, honey and water analysis.

P-55   The Analysis of Two Classes of Persistant Organic Pollutants in Challenging Edible Oil Samples

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Persistant organic pollutants (POPs) are lipophilic, and can collect in the fatty tissues of living organisms. For this reason, they bioaccumulate through the food chain and can be found in edible oils and oil-containing foods. Testing oily samples for POPs can be problematic due to the high background created by the fats present. Common extraction techniques such as liquid-liquid extraction use nonpolar solvents, which result in large amounts of fatty matrix being coextracted with the analytes. As a result, clean up using large solid phase extraction (SPE) cartridges of silica or alumina is often required for these types of samples prior to chromatographic analysis. This work will present an extraction method for PCBs and PAHs from edible oil samples using a new dual-layer SPE cartridge. Specifically, the method will be applied to the extraction of polychlorinated biphenyls (PCBs) and polynuclear aromatic hydrocarbons (PAHs) from soybean oil. Undiluted soybean oil sample was weighed directly onto the cartridge, and elution of the analytes was performed in a single step using acetonitrile. The extracts were concentrated and analyzed directly by GC/ECD for PCBs or HPLC/FLD for PAHs. The analysis data indicated excellent recoveries (above 80%) and reproducibilies (below 15%) of spiked replicates at 10 ppb.

P-56   Analysis of Bisphenol A in Milk and Canned Broths Using Molecularly Imprinted Polymer SPE and LC with Fluorescence Detection

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Bisphenol A (BPA) is used in the manufacture of many plastics and epoxy resins that are used to make milk containers and liners to seal metal food containers. Over time some of the free BPA will be leaching out from the plastics and resins to the food it contacts. Because of BPA's potential to act as an endocrine disrupter at low concentrations in humans, regulations have been enacted regarding BPA concentrations in food and beverages sold for human consumption, and associated methods for the detection and quantification of BPA in food and beverages have been developed.

This work presents a method for detection of low levels of BPA in milk and canned broths. Quantifying low levels of BPA in complex sample matrices, such as food samples, can present sample preparation challenges. Molecularly Imprinted Polymer (MIP) Solid Phase Extraction (SPE) is highly selective and considered the top choice for sample cleanup of a single compound or a class of structurally related compounds from difficult matrices. This method uses a BPA MIP SPE to extract and pre-concentrate BPA. Analysis was performed using LC with fluorescence detection. Recovery of BPA from milk at 1 ng/mL was over 80%, and from chicken broth spiked at 60 ng/mL was over 70% with good reproducibility. An unfortified beef broth was also analyzed and found to contain BPA at level greater than 60 ng/mL. We will discuss these results and address some practical issues related to the analyses.

P-57   Use of Graphitized Carbon Black and other Adsorbents for the Removal of Pigments during QuEChERS

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The clean-up of fruit and vegetable samples for pesticide analysis is complicated by the presence of plant pigments in the matrix. Pigment molecules are generally large, non-volatile compounds that tend to get trapped in the inlet liner of the GC/MS. Use of the QuEChERS clean-up method does not necessarily remove all pigments from the matrix. Method EN

15662 recommends the addition of Graphitized Carbon Black (GCB) to remove plant pigments from samples. In addition to removing plant pigments, GCBs also retain some pesticides, especially planar pesticides.   A range of plant pigments was chosen to be representative of the pigments found in various fruits and vegetables. In addition to GCB, other adsorbents were used to remove the pigments from acetonitrile solutions designed to simulate dispersive SPE extracts. As expected, the ENVI Carb removed the plant pigments very well with the exception of the highly water soluble betanin. SupelSphere displayed a similar pattern with the exception of crocin.   ZSep+ removed between 40% and 80% of the plant pigment from the solutions. Surprisingly, ZSep was poor at retaining all of the pigments with the exception of crocin.

P-58   Analysis of Antioxidants in foods and Dietary Supplements Using HPLC with Post-Column Derivatization

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Antioxidants protect cells from the damaging effects of Free radicals and offer numerous benefits for human health. Many phenolic compounds found in plants, as well as some vitamins, exhibit antioxidant activity. Several colormetric assays exist to measure the total antioxidant capacity, typically expressed as Trolox or Gallic acid equivalents.

An increased interest in antioxidants has created a demand for methods that are not only capable of determining the total antioxidant activity of the sample, but also are able to identify and quantify individual compounds known for their biological benefits. Our method abstract demonstrates that the well-know colormetic reagents, such as Folin-Ciocalteu and 2,2' – azinobis(3-ethylbensolthiazonline)-6-sulfonate (ABTS), can be successfully used for analysis of antioxidants in foods and dietary supplements by HPLC with post-column derivatization.

P-59   Modern State of GC-MS/MS Pesticide Analysis using Thermo Scientific™ TSQ™ 8000 Evo and Chromeleon™ 7.2 SR2 CDS

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Triple quadrupole mass spectrometers such as Thermo Scientific™ TSQ™ 8000 Evo GC-MS/MS systems have gained popularity over their single quadrupole counterparts because of their high selectivity and lower detection limits, especially in complex matrices such as those encountered in pesticide analysis in food. In this poster we present results of GC-MS/MS analysis of pesticides using timed (with respect to GC peak retention time) reaction monitoring (t-SRM) and show the advantages it holds over traditional segmented SRM technique. Optimized dwell times in t-SRM result in higher sensitivity when screening and/or quantitating several hundred pesticides in a single injection. The timed mode of MS acquisition combined with the Enhanced Velocity Optics (EvoCell collision cell) present in the TSQ 8000 Evo enables us to monitor several confirming transitions per analyte for a more confident confirmation without compromising quantitation sensitivity. The results we show were obtained using Thermo Scientific™ Dionex™ Chromeleon™ 7.2 SR2 CDS software, which combines powerful data analysis capability with easy pesticide analysis method creation. The Chromeleon software pesticide analyzer database contains retention times and transitions for over 1000 pesticides and other compounds of environmental interest. Finding MS/MS transitions for compounds used to be arduous and time-consuming process prone to operator error. The poster will also highlight the power of AutoSRM which is a tool for getting optimized transitions for compounds that are not present in the database. AutoSRM simplifies the process of obtaining transitions for unknown compounds with minimal user interaction.

P-60   Determination of Meat Authenticity using Peptide Biomarkers and High-Resolution Mass Spectrometry

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In recent years, we witnessed the internationalization of food production, distribution and retailing. Interestingly,

we observed a significant increase in food fraud, ranging from false label claims to the use of additives and fillers, to increase profitability. Recently in 2013, horse and pig DNA were detected in beef products sold from several retailers. Contaminated meat products are not only misleading consumers but have ethical and health implications. Mass spectrometry has become the workhorse in protein research. The detection of marker proteins could serve for both animal species and tissue authentication. Meat species authenticity was performed using a well-defined proteogenomic annotation, carefully chosen surrogate tryptic peptides and analysis using a Hybrid Quadrupole-OrbitrapTM mass spectrometer. Identification of biomarker proteins representative of a particular species has been performed. Myoglobin has 153 amino acid residues and can be methodically analyzed in silico in order to generate tryptic peptide mass lists and theoretical MS/MS spectra. Following a comprehensive bottom-up proteomic analysis, we were able to detect and identify a very specific myoglobin tryptic peptide [119-133] for each species with observed m/z below 1.3 ppm compared to theoretical m/z. Additionally, high-resolution MS/MS spectra reveal b/y ions compatible with amino acid sequence of each targeted peptides. Other myoglobin signatures peptides for pork and horse meats were detected and identified including myoglobin tryptic peptides [1-16], [80-96] and [103-118].

P-61 EPA Method 557 Quantitation of Haloacetic Acids, Bromate and Dalapon in Drinking Water Using Ion Chromatography and Tandem Mass Spectrometry

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Haloacetic Acids (HAAs) can be formed during drinking water purification in municipal water supplies during the chlorination, ozonation or chloramination of water. Reactions between chlorine and organic matter present in the water can create HAAs. There are health concerns regarding human consumption of HAAs. Because of these concerns, the US EPA has published Method 557 for the quantitation of HAAs using Ion Chromatography coupled to Tandem Mass Spectrometry (IC-MS/MS). Previous techniques for the analysis of HAAs included derivatization of the HAAs and analysis by GC-MS. The following HAAs were analyzed, along with bromate and the pesticide dalapon: Bromochloroacetic acid (BCAA), Bromodichloroacetic acid (BDCAA), Chlorodibromoacetic acid (CDBAA), Dibromoacetic acid (DBAA), Dichloroacetic acid (DCAA), Monobromoacetic acid (MBAA), Monochloroacetic acid (MCAA), Tribromoacetic acid (TBAA) and Trichloroacetic acid (TCAA). While US regulations currently only require the monitoring of 5 of the HAAs, (MCAA, DCAA, TCAA, MBAA and DBAA), interest is growing in the additional 4 HAAs and were included in this analysis. Drinking water samples were tested using San Jose, CA municipal drinking water as well as bottled water for a variety of manufacturers to test for the presence of HAAs. The response of the HAAs, bromate, and dalapon over the concentration range was linear. An instrument detection limit (IDL) was calculated for each analyte based on replicate injections and the student's t-test.

P-62 Screening and Quantitation of Micropollutants from Sewage Water in the Process of Bank Filtration using UHPLC-HRMS

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In the city of Berlin, Germany, drinking water is completely derived from ground water containing large portions river bank filtrated water. In this study, the barrier function of bank filtration for micropollutants was investigated along a transect at the lake "Tegeler See" in the city of Berlin. The transect consisted of multiple ground water sampling sites between the lake and a water supply well.

The study was performed on an EQuan Max Plus online SPE and chromatography system, coupled to a Q Exactive Focus mass spectrometer (both Thermo Fisher Scientific). 1 mL of sample was injected, preconcentrated and subsequently separated on an analytical column, resulting in a full chromatographic cycle of 15 minutes. For suspect screening, a home-built database with more than 2000 entries was applied, using isotope pattern and fragment matching for identification. Online databases like STOFF-IDENT and mzCloud were used for compound identification in the non-target screening approach.

The suspect screening yielded 93 detects, of which 60 were detected for the first time in the area of survey. Of these 32 were human drugs, 9 drug metabolites, 2 pesticides, 3 pesticide metabolites and 14 other contaminants. Quantitation

was done for 58 components, 21 of them being new detects. While in the lake 46 components were above detection limit, still 31 of them could be detected in the water supply well. 11 components were exceeding regulatory limits.

P-63   Determination of a Single Methodology for the Analysis and Quantitation of Multi-class Veterinary Drugs in Different Animal Matrices used for Consumption

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The quantification of large numbers of multi-class veterinary drugs from different meat products usually involves separate extraction methods for each matrix and/or compound class, either with SPE or LLE extraction. In addition, multiple chromatographic and mass spectrometric methods are often required, all leading to substantial preparation and analytical run time. A new method, utilizing a single chromatography run and a triple quadrupole mass spectrometer is described in this poster. Over 200 veterinary drugs can be analyzed using this robust methodology and in compliance with regulatory guidelines. To test the assay, standard curves with seven points were prepared in different matrices covering the range 10 pg/mL (ppt) to1 µg/mL (ppm). Two ions were monitored, one for quantitation and the other one for qualification. The calibration curves were linear over the ranges described.. The columns showed no deterioration in quality or performance when analyzing the different matrices one after the other. To maximize instrument robustness, a divert valve and sweep gas was utilized to prevent contamination of the ion source.

P-64   Quantitative and Qualitative Confirmation of Pesticides in Beet Extract Using High Resolution Accurate Mass (HRAM) Mass Spectrometry

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As world agricultural trade has expanded and concerns over food safety have grown, the enforcement of stricter pesticide residue regulations has become of utmost importance. The European Union (EU) and the Japanese regulations are amongst the most stringent in the world and have fueled the need for faster and more sensitive analytical methods for cost-efficent, high-throughput screening and quantitation of multi-class pesiticide residues. Here we will describe a methodology that uses high resolution accurate mass (HRAM) mass sprectrometry to quantify and confirm in a single experiment. To test the assay, standard curves with seven points were prepared in beet matrix covering the range 0.01ug/kg to 100 ug/kg. Quantitation was done on full scan parent ions while qualification was done by fragment ions (ms/ms) as well as spectra library matching. The calibration curves were linear over the ranges described. The HPLC column showed no deterioration in quality or performance when analyzing the beet matrix over multiple runs. Detection limits were well below the EU set maximum residue limits (MRL) and the R^2 values were better than 0.99.

P-65   Using Capillary IC with Suppressed Conductivity and Charge Detection to Profile Organic Acids in Juices and Beverages

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Profiling organic acids in fruit juices is important to the beverage industry to characterize flavor, maintain product quality, and to meet labeling requirements. Organic acid profiles are characteristic of the fruit source, which can identify adulteration of a more expensive product with that of a lower cost product. Microbial activity can result in undesirable and unpalatable organic acids associated with spoilage. To analyze organic acids and anions of strong acids (chloride and sulfate), which are also present in beverages, ion chromatography with suppressed conductivity is the ideal analytical method. Unlike the anions of strong acids which are fully ionized, organic acids are weakly ionized and can exhibit lower conductivity responses versus concentration than the strongly ionized anions. The Thermo Scientific Dionex QD Charge Detector promotes complete disassociation of many weakly disassociated compounds by drawing a current at a fixed potential. As a result, the charge responses of doubly-charged and triply-charged organic acids are relatively higher than conductivity.
Here we demonstrate separations of organic acids on a 4 µm particle size, capillary anion-exchange column. Four µm particle columns produce high efficiency separations, but also have higher system backpressure and therefore may

require the use of a high-pressure capillary IC system. Capillary IC, at µL/min flow rates, is always on and ready for analysis and requires only 5.2 L/yr of deionized water. The results show comparably higher QD response for organic acids as compared to chloride and sulfate. Additionally, use of CD/QD ratios to assess peak purity is demonstrated, thereby improving reporting accuracy.

P-66   The Use of XAD-2 Resin Packets For Passive Air Sampling of Chlorinated Pesticides

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XAD-2 resin is an absorbent polymeric resin often employed in the monitoring of environmental pollutants from manufacturing processes and energy production. The styrene-divinylbenzene copolymer is excellent at absorbing many semi-volatile organic chemicals including PAHs, PCBs, Chlorinated Dioxins and Furans, and many pesticides. As XAD-2 was designed for continuous sampling of gas streams, this resin can be utilized as a passive sampler to measure exposure to chlorinated pesticides in the air. XAD-2 packets were prepared using two grams of XAD-2 resin enclosed in lint-free laboratory tissue. The XAD-2 packets were deployed for thirty days in rural and urban houses in northern and central California. The concentrations of thirty one chlorinated legacy pesticides were analyzed with high resolution chromatography coupled with high resolution mass spectrometry (HRGC/HRMS) following a modified EPA method 1699. The concentrations of the pesticides were measured in pg/sample. Both urban and rural samples displayed positive results for certain chlorinated pesticides in every sample. The data also show a significant difference in concentration between rural and urban areas as well as different weathering patterns.

P-67   Analysis of EU 15+1 Priority Polycyclic Aromatic Hydrocarbons in Yerba Mate Tea Using Modified QuEChERS, Solid Phase Extraction and Gas Chromatography Time-of-Flight Mass Spectrometry

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Polycyclic aromatic hydrocarbons (PAHs) are toxic compounds found in some foods, especially those that are smoked, roasted, grilled, or dried during preparation. The European Food Safety Authority (EFSA) established a list of 15+1 priority PAHs to be monitored in food due to their potential carcinogenetic effects. PAH analysis is challenging because there are isobaric PAHs that interfere with these priority PAHs of interest making accurate quantitation difficult. Even when focusing on a small subset of the 15+1 priority list, the PAH4 and PAH8, it is important to consider non-target isobaric interferences that can skew toxicity determination.
Yerba mate tea has been growing in popularity due to the reputation of providing numerous health benefits, including increased energy and weight loss, and treatment of many health problems from headaches to hypertension. However, high incidence of esophageal cancer in populations with high mate tea consumption suggests a possible link between mate and cancer. One important consideration is the relatively high levels of toxic PAHs in mate tea, likely due to the drying process. While classic sample extraction methods yield excellent results for PAHs in tea, these techniques are time consuming and costly. A much less resource-intensive modified QuEChERS extraction and silica solid phase extraction (SPE) sample cleanup method was developed and yielded good quantitative recoveries for PAHs in yerba mate tea. Chromatographic separation of all EU 15+1 priority PAHs and their isobaric interferences were optimized on a high-phenyl stationary phase GC column. Quantitation of incurred PAHs was determined via GC-TOFMS with hydrogen carrier gas.

P-68   Optimization of Gas Chromatographic Parameters using Chromatographic Modelling Program for Halogenated Disinfection By-Products Analysis

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We present here a novel approach for optimizing the gas chromatography (GC) separation of 13 drinking water disinfection by-products (DBPs). The 13 DBPs are organochlorine and organobromine compounds commonly referred

to as the haloacetaldehydes (HAs) and haloacetonitriles (HANs). These compounds arise from chlorine based water treatment processes and are characterized by their low boiling points and similar chemical structures and mass spectrometric fragments. It is difficult to separate them on a single GC column and identify them using mass spectrometry; as a result, they have been analysed using dual-column separation followed by dual ECD analysis (US EPA Method 551). Dual column analysis is tedious in terms of equipment maintenance and data interpretation. The ability to achieve chromatographic separation of these compounds using a single GC column becomes imperative for the development of a good analytical method. Continued developments in GC stationary phase chemistry offer more choices than the methyl and cyanopropylphenyl columns recommended by EPA Method 551. Utilizing a specialized computer modelling program, we simulated the separation on four different stationary phases to achieve optimized resolution of these 13 DBPs.

Without computer assistance this optimization process would have involved numerous GC analyses and taken weeks to complete for each column excluding column changes and conditioning time. Results obtained from the modelling program as well as the validation of the analytical column will be presented. Concurrent Solvent Recondensation – Large Volume Splitless Injection (CSR_LVSI) was used to allow detection of very low levels of the HAs and HANs.

P-69   Shoot-and-Dilute GC-ECD for Analysis of Problematic Pesticides

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In gas chromatography, most of the problems with analysis occur on the front end, at the GC inlet, where compounds like Captan and Folpet and DDT and Deltamethrin can degrade during hot splitless injection, active compounds like Methamidophos and Acephate and Omethoate can be irreversibly adsorbed to inlet liner surfaces, and nonvolatile material from dirty samples can compromise the transfer of less volatile compounds of interest like heavier synthetic pyrethroids from the inlet to the GC column. These issues are magnified due to the very slow inlet flow during splitless injection, which is typically less than 2 mL/min.

A way to mitigate the problems listed above is to instead use split injection, what we term "shoot-and-dilute", where the much higher flow rate through the inlet results in a substantially reduced residence time and a higher effective transfer for difficult compounds of interest. This technique is especially appropriate with ultra-sensitive detectors like the electron capture detector (ECD). This paper will demonstrate the use of split injection with an inlet liner specifically designed for accurate and repeatable transfer of pesticides with a wide volatility and chemical class range. "Shoot-and-dilute" fast GC-ECD for trace analysis of organochlorine pesticides (including Captan and Folpet), will be demonstrated, while illustrating benefits such as increased system uptime and shorter overall analysis time and higher sample throughput.

P-70   GC-MS/MS Method Development Strategies and Lessons Learned for Multiresidue Pesticides in Food Matrices

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Gas chromatography - tandem mass spectrometry (GC-MS/MS) has enjoyed resurgence in applied markets in recent years. GC-MS/MS provides increased selectivity and sensitivity compared to GC-MS making it ideal for pesticide residue analysis. Development of robust single reaction monitoring (SRM) methods can be challenging though due to the sheer number of precursor ion options for some compounds and the lack of good precursor ions for other compounds. Another problem is finding a number of sufficiently unique MS/MS transitions to support unequivocal compound identification, especially in more complex food matrices. For this work, a single method was developed for over 200 pesticides so analyte to analyte SRM interferences as well as matrix interferences have to be considered. Organophosphorus, organochlorine, organonitrogen, pyrethroids and herbicide methyl esters are represented.

For this work, gas chromatography conditions based on efficiency-optimized flow and optimal heating rate were used to distribute the pesticides somewhat equally throughout a chromatogram to better manage the interference possibilities. Careful full-scan spectra review of closely eluting compounds was performed to reveal potential SRM conflicts prior to MS/MS experiments. SRM ion ratios for quantification and qualification ions were then determined in pesticide standard subsets, followed by an evaluation that included 203 organophosphorus, organochlorine, organonitrogen, and synthetic pyrethroid pesticides analyzed together in one GC-MS/MS run.   From the experiments done, four basic steps for SRM method development will be recommended in the poster.

P-71 Liquid Chromatographic Methods for Pesticide Analysis and Cannabinoid Profiling in Cannabis

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Cannabis for both medical and recreational purposes is gaining wider acceptance in the United States, with 23 states and the District of Columbia currently allowing some form of cannabis use. Given the potential medical benefits and changing public opinion regarding cannabis legalization, the medical cannabis industry may continue to expand across the nation. Especially for medical cannabis that is being administered to the elderly and children, product quality and safety is paramount. Currently, the suite of tests for cannabis safety is relatively well-established, however these tests are being performed with differing methods and levels of sophistication across the nation. Optimized liquid chromatography (LC) methods for cannabis will be discussed in this poster.

Because cannabis is a very high-value crop, pest and fungus control are important for both indoor and outdoor grow operations. Due to the complex nature of cannabis matrix, tandem MS techniques will be required for pesticide analysis. Extraction methods for reduction of ion suppression and optimized analysis conditions will be presented. Effects of high levels of co-eluting cannabinoids will also be explored.

One of the most common LC methods for medical cannabis is potency testing. Accurate potency results are essential for medical cannabis for dosing purposes. Using an optimized method and a LC-UV instrument can provide fast analysis of all common cannabinoids found in cannabis. While potency can be analyzed via gas chromatography (GC), LC allows easy quantification of both the acid and neutral forms of cannabinoids, which is important for edible products that are quickly gaining popularity.

P-72 Gas Chromatographic Methods for Analysis of Pesticides and Residual Solvents in Cannabis and Cannabis Concentrates

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Over the course of the past several years, use of cannabis for medical purposes has gained acceptance in the US. Both medical and recreational cannabis are now available in a wide variety of alternative dosage forms, from vaporizers to edible tinctures. Medical users of cannabis often prefer dosage forms that are not smoked or are vaporized. Many alternative dosage forms are now manufactured using cannabis concentrates. Cannabis concentrates are produced via either solvent or supercritical fluid extraction of cannabis plant material. While concentrates offer a more consistent manufacturing solution for alternative dosage forms, any co-extracted contaminants from the plant material will also be concentrated, and residual solvents also become a concern.

Co-extracted pesticides in cannabis concentrates are a cause for concern from a consumer safety standpoint, especially for consumption through vaporization. Due to the complex nature of cannabis, multidimensional GC or tandem MS analysis is required for the detection of pesticides in this matrix. Common pesticides used on cannabis include pyrethrins, diazanon, and bifenthrin. Additionally, high levels of interfering cannabinoids pose an analytical challenge. Extraction and clean-up techniques for cannabis and cannabis concentrates will be explored.

In addition to co-extracted pesticides, residual solvents may be present in cannabis concentrates, depending on the extraction method used to produce the concentrate. Currently, several states have regulations in place for residual solvent testing, and laboratories are beginning routine testing for residual solvents. A fast, easy, full evaporation technique-headspace-GC method for quantification of low level residual solvents in cannabis concentrates will be presented.

P-73 Impact of Analytical Reporting Criteria on MRLs and Risk Assessment

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The criteria utilized for reporting pesticide residue analytical data less than the limit of quantitation (LOQ) can vary

widely across laboratories, geographies and government agencies. These differences can cause downstream differences in interpretation of non-numerical results (censored data), and thus non-harmonization in the calculation/setting of MRLs and risk assessment determinations. There are various options for reporting residue results less than the LOQ, including <LOQ, one-half the LOQ, or a numerical value. In addition, analytical values below the LOD, could be reported as <LOD, one-half the LOD, or ND. When these results are carried further into calculations of Sum, Mean, Median, etc., the interpretation of the reported values become important and can lead to different outcomes. Examples are provided to represent the impact on MRLs and risk assessments and demonstrate that utilizing numerical values for residues less than the LOQ when possible will increase accuracy of interpretations. Thus, harmonization in the reporting of the analytical data below the LOQ will lead to more consistency and accuracy in interpretations of the data.

P-74   Quantitative Determination of a Pesticide and Its metabolites in Soil with Sonication Extraction

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An analytical method was developed to quantitatively determine the residues of a pesticide and its metabolites in soil as part of the terrestrial field dissipation study to understand the mobility and persistence of these molecules in the environment.

The complexity of method was highlighted by the need to optimize the HPLC conditions for the diverse group of analytes as well as the requirement for an appropriate extraction procedure. For the HPLC separation, the parent and its metabolites were distributed over a wide range of polarities, which posted a challenge in separation. This was overcome by using a specific analytical column capable of retaining both polar and non-polar analytes. For the extraction, conditions needed to be harsh enough to efficiently extract the tightly bound terminal degradates, but also suitable enough without breaking down the parent and early metabolites. Both Accelerated Solvent Extraction (ASE) and sonication extraction demonstrated sufficient extraction efficiency. However, the sonication extraction was eventually used in the method due to its superior efficiency and throughput. Statistics will be shown to demonstrate the method performance.

P-75   Doing More with Less – The Advantages of Miniaturization and High-Throughput

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Traditionally, crop method design has been based on 10-20 gram subsamples extracted in 10-20X mL of solvent in 125-250 mL poly bottles. Samples are processed by maceration for 3-5 minutes on a per/sample basis. Further procedures like centrifugation, LLE, SPE, and filtration are performed with limited capacity. Final fractions are individually vialed for analysis either by GC-MS or LC-MS/MS. Processing a sample by grinding with dry ice or cryogenic grinding (Freezer Mill-Liquid Nitrogen) ensures a representative homogeneous sample down to 50-100 milligrams. The miniaturized sample can now be processed using formats like 2 mL tubes and titer plates. Simultaneous processing of the samples can be performed using High-Throughput components like the Geno/Grinder tissue homogenizer, QuEChERS Technique, high capacity centrifugation, SPE and filtration using titer plates. High-Throughput techniques allow more samples to be processed in less time. Faster sample analysis time is advantageous for companies using the outsourcing model, data submission, or where limited capacity at a CRO is encountered. Earlier decisions can be made due to decreased study length and availability of residue data when compared to traditional sample processing requiring 5X more time producing the same data points. Reduced sample size promotes Lean Lab – reduction in solvent consumption, reduction in waste stream, reduction in storage while increasing efficiency and throughput.

P-76   Level of 2,4-D or Dicamba Residue Found in Cucurbit Fruit from a Simulated Drift Scenario using LC/MS/MS

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Vegetable production in Georgia has a farm gate value of nearly $1 billion including 33 vegetables produced for fresh

market; 32 of which are sensitive to 2,4-D and dicamba. Cantaloupe and cucumber experiments were conducted during 2014; cantaloupe in the spring and cucumber in the fall at the Tifton Vegetable Park. Plots were 12 feet wide by 20 feet long with transplants planted into a 32-inch wide by 8-inch tall raised bed. Cucurbits were treated with 2,4-D amine or Clarity (dicamba) at 1/75X or 1/250X rate during three growth stages. During harvest, two fruit from each plot were bagged and delivered to The Georgia Department of Agriculture for residue analysis. The pesticide residues laboratory prepped, extracted, and analyzed the samples using modified QuEChERS method and API 3200 LC/MS/MS. The residue levels detected by the Georgia Department of Agriculture were extremely consistent within treatments. For example, dicamba levels detected in cantaloupe at the 1/75X rate 18 DBH (days before harvest) by replication were as follows: 0.01, 0.02, 0.015, and 0.011. For cucumber, dicamba levels detected at the 1/25X rate 7 DBH by replication were as follows: 0.004, 0.01, 0.01, and 0.005.

P-77   Sandwich Injection Method for Pesticide Analysis by Agilent 7000 GCMSMS System

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The Chemical Residue Laboratory of the Florida Department of Agriculture analyzes fruit and vegetable samples for pesticide residues. The analysis has evolved over time and the extraction procedures have been simplified. The use of QuEChERS techniques for extraction is prevalent in pesticide residue labs today. However the extracts are dirty and present challenges to the GC and mass spectrometer systems. One remedy is to utilize an analyte protectant solution to assist with matrix interactions in the GC systems. In order to accomplish this, the CR Lab uses a Gerstel MPS to add the protectant to the sample extracts. We have come up with an alternate method to accomplish this task, which reduces time as well as human error. This sandwich injection method can be run with the 7693A Automatic Liquid Sampler provided with the Agilent 7000 GCQQQ. The sandwich injection method prepares each sample extract and standard-in-matrix very precisely and is very effective. The implementation of this technique provides a quick preparation of the extracts for analysis, and ensures a well maintained GC with little degradation of analytes. This poster will illustrate the pros and cons for the technique versus manual preparation.

P-78   Adduct Interferences Found in Large Pesticide Residue Screens Using High Resolution Mass Spectrometry

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At the Florida Department of Agriculture we use ultra high performance liquid chromatography (UHPLC) and high resolution mass spectrometry (HRMS) to screen for 226 pesticides. While the combination of UHPLC and high resolution full scans that include two fragmentation experiments has proven to be an effective analytical technique in overcoming most interferences from molecular formulas and column chemistry; throughout method development to increase our screening list to nearly 500 compounds and their fragments, we have found large screens require attention to potential adduct interferences. For example, in our half-hour LC method, demeton-S has a retention time of 11.59 minutes and buturon elutes at 11.87 minutes. In addition to a nearly 18 second difference in retention time due to a relatively long chromatography method, this would not appear to be a problem because of the drastic difference in mass between the two compounds; with an H^+ adduct, demeton-S and buturon have an m/z of 259.0586 and 237.0795, respectively. However, if buturon has a Na^+ adduct instead of an H^+ adduct, its m/z is 259.0609 and appears quite similar to demeton-S, even for UHPLC –HRMS systems. Our laboratory has discovered this potential problem and has taken various steps to avoid false positives such as these in preparation for our upcoming method validation.

P-79   A Rapid Liquid Chromatography Determination of Formaldehyde in Cod

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A rapid method for the determination of free formaldehyde in cod is described. It uses a simple water extraction and extracted formaldehyde is derivatized with 2,4-dinitrophenylhydrazine (DNPH) to form a sensitive and specific chromophore for HPLC detection. Although this formaldehyde derivative has been widely used in past tissue analysis,

this paper describes an improved derivatization procedure. The formation of the DNPH formaldehyde derivative has been shortened from greater than 1 hour to 2 minutes and a stabilizing buffer has been added to the derivative to increase its stability. The average recovery of free formaldehyde in spiked cod was 63% with an RSD of 15% over the range of 25-200 ppm (n= 48). The HPLC procedure described here was also compared to a commercial qualitative procedure- a swab test for the determination of free formaldehyde in fish. Several positive samples were compared by both methods.

P-80   Strategies for the Application of HRMS for the Semi-targeted Analysis of Veterinary Drug Residues in Aquacultured Products

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High resolution mass spectrometry (HRMS) acquires data in a nontargeted manner, potentially allowing for the detection of any compound in the sample. Data analysis can either focus on specific (target) residues or look for unexpected (nontarget) analytes. "Semi-targeted" data analysis searches HRMS data against a large database of known compounds. The objective of this work is to evaluate the application of HRMS for screening veterinary drugs and other chemical contaminants in fish and other aquacultured products. Initial work was performed using a LC Q-TOF MS instrument. This proved to be a valuable tool for finding additional residues in aquaculture beyond what was expected, but challenges remained including the need to evaluate numerous false positives. Various data acquisition and analysis programs are now being evaluated using a Q-Exactive instrument. Different strategies for sample extraction, method validation, and data analysis are being investigated. Data independent (DIA) and data dependent (DD-MS^2) analysis to collect fragment ion data for both targeted and nontargeted analytes was performed. This approach has successfully detected and confirmed the identity of residues in fish at ng/g levels using accurate mass data, retention times, fragment ions, and isotope patterns. HRMS is an emerging analytical technology that will expand the scope of residue monitoring.

P-81   Direct Determination of Glyphosate, Glufosinate, and AMPA in Milk by Liquid Chromatography/Tandem Mass Spectrometer

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A simple high-throughput liquid chromatography/tandem mass spectrometry (LC/MS–MS) method was developed for the determination of glyphosate, aminomethylphosphonic acid (AMPA) and glufosinate in milk using a reversed-phase with weak anion-exchange and cation-exchange mixed-mode Acclaim™ Trinity™ Q1 column. One milliliter of milk was shaken with three milliliters of water containing Na_2EDTA and acetic acid to precipitate protein for 10 min. After the centrifugation, the supernatant was passed thru an Oasis HLB SPE to retain suspended particulates and phospholipids. The sample was directly injected and analyzed in 6 min with no sample concentration or derivatization steps. Two multiple reaction monitoring (MRM) channels were monitored in the method for each target compound to achieve true positive identification. Three internal standards corresponding to each analyte were used to counter matrix suppression effect. Linearity of the detector response with a minimum coefficient of determination (R^2) value of more than 0.995 was demonstrated in the range of 2 to 1000 ng/mL for each analyte. By using standard in solvent with internal standard calibration method, average recovery for all analytes at 0.025, 0.1, 0.5, and 2 ppm (n = 7) are between 84-111% with a relative standard deviation of less than 8%.

P-82   Direct Determination of Glyphosate, Glufosinate, and AMPA in Soybean and Corn by Liquid Chromatography/ Tandem Mass Spectrometer

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A simple high-throughput liquid chromatography/tandem mass spectrometry (LC/MS) method was developed for the determination of glyphosate, glufosinate and aminomethylphosphonic acid (AMPA) in soybean and corn using a reversed-phase with weak anion-exchange and cation-exchange mixed-mode Acclaim™ Trinity™ Q1 column. Two grams of sample was shaken with ten milliliters of water containing ethylenediaminetetraacetic acid disodium salt (Na_2EDTA)

and acetic acid for 10 min to precipitate protein. 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 with no sample concentration or derivatization steps. Two multiple reaction monitoring (MRM) channels were monitored in the method for each target compound to achieve true positive identification. Three internal standards corresponding to each analyte were used to counter matrix suppression effect. Linearity of the detector response with a minimum coefficient of determination (R^2) of more than 0.995 was demonstrated in the range of 2 to 1000 ng/mL for each analyte.

P-83   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. Alternative methods were investigated, including derivatization of 3-MCPD using phenylboronic acid and headspace analysis for 1,3 DCP. Additionally, a new technique using microvial thermal desorption coupled with GCMS was developed involving minimal sample preparation and the rapid assessment of 3-MCPD contamination in soy sauce samples.

P-84   Comparison of Multiple Methods for the Determination of Sulfite in *Allium* and *Brassica* Vegetables

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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 method (MW) produces false positive results with vegetables from the *Allium* (garlic) and *Brassica* (cabbage) genera due to the extraction conditions causing endogenous sulfur compounds to release SO_2. Because of this bias, special consideration is needed for regulatory analyses. Recently, modifications to the MW method were published that have reduced this false positive in garlic. However, no other vegetables from these genera have been investigated. In addition, an LC-MS/MS method was developed for sulfite analysis but has not yet been tested with these problematic matrices. Eleven vegetables were selected from these genera and were analyzed using four sulfite methods to determine the false positive rate.   Sulfite concentrations greater than 10 ppm SO_2 were observed with the MW analyses. The modified MW method reduced the concentration to 10 ppm SO_2 or below for all matrices analyzed. The LC-MS/MS method had concentrations below 10 ppm for the *Brassica* samples but was not successful with the *Allium* matrices. Alternate LC-MS/MS sample preparation steps were investigated to determine if this false positive could be reduced.   The ability to eliminate false positives will enable accurate determination of added sulfite to ensure compliance with sulfite labeling requirements.

P-85   GC-MS/MS Determination of Pesticides and Tobacco-Specific Nitrosamines (TSNAs) in Finished Cigarette Tobacco Using a Modified QuEChERS Method

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A number of brand-name imported cigarettes suspected to be counterfeit have been evaluated by the Forensic Chemistry Center (FCC) using multiple screening methods. A multi-residue pesticide method for finished cigarette tobacco was developed using QuEChERS extraction followed by LC- or GC-MS/MS analysis. A commodity and risk-based approach was used to streamline the number of pesticides targeted by this procedure. For this study, GC-MS/MS with multiple reaction monitoring (MRM) was utilized for the identification and determination of 56 commonly encountered pesticides and selected TSNAs in finished cigarette cut filler. The GC-MS/MS procedure compliments LC-MS/MS methods by providing critical coverage for most non-polar pesticides that are not amenable to electrospray ionization (ESI) in the positive mode. A novel solvent exchange step is employed to achieve a final extract that is more amenable to GC-MS. Sample composites were prepared for analysis by removing cut filler from a designated number of cigarettes followed by cryogenic milling to achieve homogenization. Preliminary validation experiments were carried out by applying the procedure to a brand of commercially available organic cigarettes, which were also used to prepare matrix matched standards. Specificity, accuracy, precision, instrumental limit of detection/quantitation (LOD/LOQ) and linearity from the initial study will be presented.

P-86   Analysis of Acid Herbicides Using Modified QuEChERS with Fast Switching ESI+/ESI- LC-MS/MS Determination

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A method for the determination of 35 acid herbicides in food matrices was developed, validated and implemented. The procedure utilizes a modified QuEChERS extraction procedure coupled with determination by liquid chromatography tandem mass spectrometry (LC-MS/MS). The acid herbicides analyzed are all organic carboxylic acids including the older chlorophenoxy acid herbicides such as 2,4-D, dicamba, 4-CPA, quinclorac, etc, and many of the newer imidazolinone herbicides such as imazethapyr, imazaquin, etc. In the procedure, 10 ml of water is added to 5 g of sample, and extracted with 25 ml of 1 % formic acid in acetonitrile for 1 minute. The acetonitrile is salted out of the extract with sodium chloride and magnesium sulfate and then centrifuged. The acetonitrile is diluted 1:1 with water for determination by LC-MS/MS that uses fast switching between positive and negative electrospray ionization (ESI) modes. Average recovery for all the compounds, excepting aminocyclopyrachlor, was 95% with a precision of 8 %. The method detection limits for all residues was less than 10 ppb and the average coefficient of determination of the method recoveries was greater than 0.99 for all but two compounds tested. The method was used successfully for the determination of acid herbicides in FDA's Total Diet Study. The procedure proved to be accurate, precise, linear, sensitive, and rugged.

P-87   Validation Study of 204 Pesticides in Fruits and Vegetables by QuEChERS and LC-MS/MS

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A multi-laboratory (n=5) 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 to a pre-determined volume prior to analysis by LC-MS/MS. All laboratories employed previously comminuted and homogenized orange, carrot and spinach (or similar vegetable) as well as two to three local produce that are unique or of interest to the participating laboratory, ABSciex iDQuant Standards Kit for Pesticide Analysis, Restek Q-Sep QuEChERS Kits and an ABSciex ABSciex Qtrap 5500 mass spectrometer. LC parameters included the use of a Restek Ultra-Aqueous C_{18} Column (100 mm x 2.1 mm, 3 μm), 10 mM NH_4HCO_2 buffer (A: water, B: methanol) with 0.1% formic acid, 0.5 mL/min flow rate with 5 μL sample injection and MS/MS parameters were optimized individually. Each sample batch prepared contained 4 matrix-matched blanks, 2 procedural blanks, 2 procedural spikes (spiked/fortified solvent samples) and 4 method spiked samples at 10, 50, 250 parts per billion (ppb).   Results from one of five participating laboratories demonstrates limit of detection, limit of quantitation and method detection limits < 0.1 ppb and R^2 > 0.995 except orange and wheat flour for which for R^2 > 0.990. Percent recovery ranges 70% < x < 110% with < 10% RSD except wheat flour which exhibited larger RSD at all spike levels . Results from all participating laboratories will be evaluated in order to understand the ruggedness of the method.

P-88   Evaluation of an Automated QuEChERS (Quick, Easy, Cheap, Effective, Rugged, Safe) Sample Preparation Workflow for Determination of Pesticides in Fresh Produce using LC-MS/MS

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The AutoMate Q-40 is a robotic system designed to optimize and automate the QuEChERS (Quick, Easy, Cheap, Effective, Rugged, Safe) sample preparation workflow. The performance of this system was evaluated in comparison to a manual sample preparation workflow using the AOAC 2007.01 unbuffered QuEChERS methodology where all samples were analyzed using liquid chromatography – tandem mass spectrometry (LC-MS/MS). Matrices used in this study were cabbage, Valencia orange, raisin and purple corn flour. A Restek LC Multiresidue Standards Kit for Pesticide Analysis, Restek Q-Sep QuEChERS Kits and an ABSciex ABSciex Qtrap 5500 mass spectrometer were also employed. LC parameters included the use of a Restek Ultra-Aqueous C_{18} Column (100 mm x 2.1 mm, 3 μm), 10 mM NH_4HCO_2 buffer (A: water, B: methanol) with 0.1% formic acid, 0.5 mL/min flow rate with 5 μL sample injection and MS/MS parameters were optimized individually. Each sample batch prepared contained 4 matrix-matched blanks, 2 procedural blanks, 2 procedural spikes (spiked/fortified solvent samples) and 4 method spiked samples at 10, 50, 200 parts per billion (ppb). Results were determined for the instrument (limit of detection, limit of quantitation and linearity) and method (method detection limit, recoveries, relative standard error and percent uncertainty at the spike level) and will be compared to determine the statistical differences (if any.)

P-89   Comparison of Q-ToF and Q-Orbitrap HR/LCMS Platforms in a High-Throughput Regulatory Setting for Determination of ~1000 Pesticides, Toxins, Drugs and Other Unexpected Food Contaminants

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There is a need for the FDA pesticide monitoring program to dramatically improve its scope of analysis for pesticides and chemical contaminants that are not included in their MS/MS targeted screening procedures which currently target < 400 compounds. In this study high resolution mass spectrometry (HRMS) is used to address this problem by simultaneously monitoring for all of the usual LC-MS/MS target compounds plus any additional compounds (provided reference standards were available) that are amenable to analysis by +ESI LC-MS/MS. Commercial databases from each vendor will be expanded to create a vast database for pesticides, metabolites, drugs, toxins and other contaminants of interest to FDA. This has the potential to include thousands of different known and/or unknown compounds. A wide variety of real-world, regulatory samples that include incurred residues are used to evaluate the two different HRMS platforms as well as compare to data from the routine MS/MS procedures. The results are used to compare the capabilities of each HRMS platform with respect to sample throughput, applicability to a variety of matrices and potentially determine which platform provides the best fit for the FDA pesticide monitoring program. Unknown and negative mode analysis will be addressed in future studies.

P-90   Optimization of Solvent Polarity for Cleanup of QuEChERS Extracts and Recovery of Pesticides that Require Determination by Gas Chromatography

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A dispersive solid phase extraction (dSPE) cleanup of QuEChERS extracts that optimizes the recovery and cleanup of pesticides and industrial chemical contaminants not amenable to analysis by LC-MS/MS has been developed for use with GC-MS/MS determination. In the study method the acetonitrile extract is diluted with ethyl acetate (1:1) during the dSPE cleanup to improve recovery of less polar compounds. Also, formic acid (0.1 %) is added to the injection vial to improve GC performance of base-sensitive analytes. Four matrices (carrot, lettuce, orange, and barley), fortified at 0, 20, 100, and 400 ng/g, were extracted and treated by the proposed method. Quantitative results of 53 representative pesticides were obtained using matrix-matched standards, with all target compounds returning greater than 69 % average recovery and an overall accuracy of 94.3 % (n=30). Method extension validation was conducted to include ~220 targets previously validated in this laboratory with excellent results. The average recovery for all spiked compounds in all four matrices was 95.6 %. Recoveries of the ~220 target compounds were also compared while using toluene and methylene chloride as co-solvents.

P-91   Multiresidue Pesticide Analysis of Teas using Ultrahigh Performance Liquid Chromatography coupled with Quadrupole-Orbitrap Mass Spectrometry

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A method was developed to detect pesticides in teas using salt-out acetonitrile extraction, solid-phase dispersive clean-up, and followed by analysis using ultrahigh-performance liquid chromatography and positive electrospray ionization-quadrupole Orbitrap high resolution mass spectrometry (UHPLC/Q-Orbitrap MS). Full scan mass spectral data was acquired for quantitation and data-dependent scan for identification and quantification. The method was validated for the determination of the limit of detection and quantitation, fortification studies of the recovery and precision at levels of 10, 25, 100 and 500 μg/kg in black and green tea matrices and the analysis of commercial tea products. The limits of detection and quantitation were analyte-and matrix-dependent and averaged in the low μg/kg for the two tea types. A mass spectral library and compound database were also developed by optimizing instrument conditions to create data-dependent MS/MS spectra and extracting their respective product ions from its precursor ion for 603 pesticides. Results show that the frequency of false negatives decreased (50% to 10%) as the pesticide concentration is increased and is attributed to instrument sensitivity and more specifically, ionization of the precursor and fragmentation of stable product ions. Commercial teas were analyzed and results reported. The UHPLC/ESI Q-Orbitrap MS demonstrated the capability to quantitate and identify pesticide residues in tea products for routine practice.

P-92   Pesticides and Persistent Organic Pollutants in CAMELLIA *SINENSIS*

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Beverages made from the infusion with boiling water of leaves from the plant Camellia *sinensis* (tea) are the most widely consumed drink in the world only exceeded by water. Tea beverages were originally and continue to be consumed for their medicinal properties as well as their aromatic flavors. Green teas have been associated with improved cardiovascular health and reduced inflammation. In 2013, FDA conducted a cross-sectional survey screen 62 black, green, white and Oolong tea products. Pesticides with no established US EPA tolerance level for tea were found in 31 tea products (50%). Two pesticides, bifenthrin and buprofesin registered for use on dried tea leaves, were also measured in 31 and 4 teas respectively. Cypermethrin, which has a 0.4 ppm tolerance on tea plant oil was found on 8 tea products. Four teas contained measurable amounts of endosulfan 1&2. Two teas had measurable amounts of either HCHs (α,β,γ,δ) or heptachlor. Ten tea products had measurable amounts of DDT-p,p', DDE-p,p' and/or DDT-o,p' with concentrations ranging 1-87 μg/kg. In nine products, DDT-p,p' dominated the isomer/metabolite pattern and one tea contained only DDE-p,p'. DDT-o,p' isomer was measured in 6 of 10 teas at lower concentrations than found for DDT-p,p'. The ratio between DDT-p,p' and DDT-o,p' (3.2-7.3) is in the approximate range found for technical DDT. The following pesticides were also identified in teas: anthraquinone, azoxystrobin, bifenthrin, buprofesin, chlorpyrifos, cyhalothrin, cypermethrin, deltamethrin, dicrotophos, fenvalerate, heptachlor, phenylphenol, pyridaben, tebuconazole, tebufenpyrad and triazophos with concentrations between 1-3200 μg/kg.

P-93   Broad Spectrum Pesticide Residue Analysis of Bee Pollen collected from the Northern Great Plains

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A pesticide broad-spectrum analysis, including 175 commonly used pesticides, of 75 pollen samples collected from the Northern Great Plains was conducted. Analyses were carried out using a combination of liquid and gas chromatographic techniques combined with tandem mass spectrometry (LC-MS/MS and GC-MS/MS). The samples were collected from areas of federal conservation lands in North Dakota (ND), which diminished from 3 million acres in 2006 to about 1.8 million acres in 2012, possibly affecting the health of bees in this area. The study is part of the Environmental Quality Incentive Program (EQIP) and the Conservation Reserve Program (CRP) led by the USDA to improve the quality of the federal conversation lands. Results of the residues obtained from this study combined with genetic sequencing of pollen DNA and crude protein analyses to identify the plants on which honey bees forage may provide information on the type and amounts of pesticides incurred in these plants to which honey bees and other pollinators are exposed.

P-94   Challenges for Analysis of Pyrethroid Residues: Overall Strategies and Solid Phase Micro-extraction (SPME) Case Study

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Pyrethroid insecticides play an important role controlling insect pests in agriculture and home and lawn care, and also in protecting humans and animals from parasites. They thus can sometimes be found at low levels in environmental samples; therefore accurate and highly sensitive analysis of pyrethroids in various matrices is important. Pyrethroids are hydrophobic and sorb very strongly from water onto any particulates as well as to the walls of the sample container. Strategies used in multi-analyte pyrethroid analyses will be presented and discussed. The quantification method of choice for pyrethroid methods is negative chemical ionization gas chromatography with mass selective detection (NCI-GC-MS). Due to observed effects of co-extracted matrix components on instrumental response (especially after multiple injections), the Pyrethroid Working Group has invested in the synthesis of stable isotope analogues, which provide an effective normalization of instrumental response, and allow successful analysis of difficult and highly variable matrices such as biosolids from wastewater treatment plants. These stable isotope standards have also proved useful as surrogates for pyrethroid analysis methods, especially for matrices (such as the aforementioned biosolids) which are variable in composition. Recent investigations using solid phase micro-extraction (SPME) with NCI-GC-MS will be highlighted. This analytical approach for aqueous samples quantifies only freely dissolved (and thus bioavailable) pyrethroids, and excludes residues adsorbed to small particles (and container walls) or complexed with dissolved organic carbon. Vessel wall adsorption is a problem, especially for calibration standards, and techniques developed to minimize these factors in the SPME analysis will be presented and discussed.

P-95   The Development and Validation of a High-Performance (HP) Lateral Flow Immunoassay (LFIA) for the Rapid Screening of a Domoic Acid (DA) from Shellfish Extracts

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Phycotoxins can have serious implications for the economy and public health. One prevalent toxin that can be present in filter-feeding marine organisms, such as shellfish, is domoic acid (DA). Clinical symptoms due to consumption of DA-contaminated shellfish may include permanent short term memory loss, disorientation, confusion, in addition to more typical food poisoning symptoms associated with the gastrointestinal tract (vomiting and diarrhoea). DA has been widely referred to as the primary amnesic shellfish poisoning (ASP) toxin, based on a characteristic neurological symptom observed as a result of the 1987 crisis in Canada; maximum permitted levels of, typically, 20 mg DA per kg shellfish tissue (20 ppm) are imposed for most species. Lateral flow immunoassays (LFIAs) are widely acknowledged for providing a low cost and simple means to rapidly screen samples. Due to these attributes, and minimal requirements for

equipment, LFIAs can be tested off-site or in remote locations. This would provide rapid results, and could potentially complement official, established marine biotoxin monitoring methods. However, performance issues associated with many LFIAs have been widely reported within the literature and include accuracy, sensitivity, interference, matrix effects, stability, ruggedness, robustness, reproducibility and lot variability. In addition to the innate strengths of the LFIA method (portable, simple, rapid, low cost), the ideal LFIA should also address the performance issues commonly encountered with the method. In this presentation, the development and validation of the first high performance-LFIA (HP-LFIA) for DA is reported, which fully meets the demands of both industrial and regulatory end users.

P-96   The Development and Validation of a High-Performance (HP) Lateral Flow Immunoassay (LFIA) for the rapid Screening of Okadaic Acid (OA) and Dinophysistoxins (DTXs) from Shellfish Extracts

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Okadaic acid (OA) group are globally occurring and regulated toxins produced by dinoflagellate species. The OA group of toxins are lipophillic, heat stable polyether toxins consisting of OA and dinophysistoxins (DTXs). Ingestion can result in diarrheic shellfish poisoning (DSP). Clinical symptoms include vomiting, nausea, abdominal cramps and diarrhoea. Typical maximum permitted levels for shellfish are 160 ug OA equivalents per kg shellfish tissue (160 ppb OA eqs). OA eqs are the sum of OA group toxins which are adjusted according to assigned toxicity equivalence factors (TEFs). This includes OA, DTX1 (35-methyl-OA), DTX2 (OA isomer) and DTX3 (which represents a wide range of acyl derivatives of OA, DTX1 or DTX2). OA and DTX1 toxins (inc. acyl derivatives) are regarded as equally toxic (TEF: 1.0), whereas DTX2 (and its acyl derivatives) are deemed less toxic (TEF: 0.6). Current confirmatory methods used by regulatory bodies for the detection of lipophillic toxins include the mouse bioassay (MBA) and LC-MS/MS procedures. A simpler and faster method is needed for screening of samples. Lateral flow immunoassays (LFIAs) provide simple means to rapidly screen samples and complement official marine biotoxin monitoring methods. Literature reported issues with LFIAs include: accuracy, sensitivity, interference, matrix effects, stability, ruggedness, robustness, cross-reactivity, reproducibility and lot variability. The ideal LFIA provides strengths (portable, simple, rapid, low cost) and addresses the performance issues commonly encountered with the method. The development and validation of the first high performance-LFIA (HP-LFIA) fully meeting the demands of both industrial and regulatory end users testing OA group is reported.

P-97   Authenticity of Spice: Detection and Quantification of Allergens in Spice using LC-MS

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Recent screening of spice has forced numerous manufacturers to recall their products due to the presence of various plant-based allergens (ie. peanut). Plant-based allergens make up half of the FDA's hit-list of priority allergens here in the USA. Allergy or intolerance to wheat, soy, peanut and tree nuts affect more than 4% of the North American population. For the detection of allergens, the food industry currently relies mainly on the ELISA assay (enzyme-linked immunosorbent assay). There are a variety of ELISA kits available that are used successfully, however, each kit targets only one analyte (ie. wheat or soy or peanut etc.) and can suffer serious drawbacks, such as lack of required sensitivity and negative/positive bias in their quantitative results. In addition, the color of some spices interferes with the wavelength used for ELISA detection, thus screening some spices is nearly impossible using this technique. To date, there has been virtually no work published on establishing the authenticity of spice using LC-MS. This presentation will discuss the development of an analytical method capable of the quantitative detection of trace levels of peptide markers representing multiple plant allergen proteins. This work utilized a proteolytic digestion sample preparation procedure, LC-QTOF accurate mass to identify representative peptide markers and MS/MS analysis of the peptides in matrix. The method was tested on a wide variety of spices and data will demonstrate how this multiplexed LC-MS assay allows accurate and sensitive detection of multiple allergens in a single assay at concentrations down to the ppb range.

P-98   Are Perfluorinated Compounds Leaching Into Our Food?

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The purpose of this project was to study the presence of PFCs (Perfluorinated Compounds) in a variety of foods that are surrounded by materials that use these compounds for moisture wicking. PFCs have been synthesized for more than 50 years and are used in numerous industrial and consumer products. They repel oil and water from clothing, carpeting, furniture, and are used in food packaging such as pizza boxes, fast-food containers, and microwave popcorn. They are also commonly used in cooking utensils as nonstick coatings.

A triple quad mass spectrometer setup with a method to look for a number of PFCs was used to analyze a variety of different food samples that come in contact with packaging thought to contain PFCs. The mass spectrometer is a powerful analytical instrument used in many labs to detect targeted analytes such as PFCs at really low levels. Samples were first weighed then extracted with methanol, analyzed by mass spectrometry and data reviewed using quantitation software.

We did not detect PFOA or PFOS (this is positive since they are banned and not produced anymore), but we detected some new replacement chemicals which are known to be used in packaging material. These newer chemical substitutes are less concerning for the environment since they degrade faster and are less persistent. However, it is not known yet if there is a negative effect on human health. Future studies are needed to investigate this. In summary, are esthetics really worth the health risk? A little mess never seemed to hurt anyone!

P-99   Quantitation of the Pesticide 1080 (Sodium Fluoroacetate) in Milk and Infant Formula

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Recent threats, in the form of letters send to farming and dairy industry leaders in New Zealand send last November, were accompanied by small packages of milk powder that were shown to contain the presence of the pesticide 1080 (Sodium Fluoroacetate). 1080 is widely used in New Zealand to protect New Zealand's native flora and fauna against introduced pests, like possum. The sender demanded that the New Zealand government stop using 1080.

There is a need to correctly identify and accurately quantify 1080 in milk and milk products to protect consumer's health and to protect the production of dairy products in New Zealand and other countries.

Here we present initial results of method development to quantify 1080 using LC-MS/MS. The SCIEX QTRAP® 4500 and 6500 system was used with Electrospray Ionization in negative polarity. Two MRM transitions were monitored to allow simultaneous quantitation and identification based on the ratio of quantifier and qualifier ion. LC separation was achieved using a normal phase setup with a HILIC column. Sample preparation and cleanup was based on a generic "QuEChERS" like protocol with hexane based fat removal.

Initial results indicate an instrument detection limit below 1 ng/mL for the Sodium Fluoroacetate. The initial method LOQ (including sample preparation and extract dilution) was determined at 10 µg/kg. Further optimization of sample preparation and the addition of an internal standard to compensate matrix effects is needed.

P-100   The Use of LC-MS/MS for the Identification of Allergens in Spices

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The prevalence of food allergies in the United States is estimated at around 6% for children and reports suggest that the number of allergies is rising. Screening for allergens is traditionally performed using enzyme-linked immunosorbent assays (ELISA). ELISA can generate variable results, and false-positive as well as false-negative results occur especially. Additionally each allergen requires a separate kit so a method that could unambiguously confirm the identification of individual allergens in a multiple allergen screen would be invaluable.

Recent findings of allergens in spices (i.e. peanut in products that contain ground cumin or cumin powder and almond in paprika and paprika containing foods) highlighted the need for analytical methods to quickly and accurately identify the presence of allergens in food to protect consumer's health.

Here we present data acquired by LC-MS/MS for the screening of multiple allergens including peanut in cumin and hazelnut in paprika. Food samples were extracted and then the allergic proteins were reduced, alkylated and digested using trypsin. The peptides from the digested proteins were purified using solid phase extraction and these extracts analyzed by LC-MS/MS and reverse phase chromatography using positive mode electrospray ionization. The mass spectrometry method utilizes the *Scheduled* MRM™ algorithm. At least 9 transitions (3 transitions per 3 peptides) were detected per allergen to enhance the confidence in identification.

P-101 Identification, Quantitation and Confirmation of Pesticides in Food Samples using Advanced LC-MS/MS Techniques

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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 a novel SCIEX QTRAP® system using Electrospray Ionization (ESI). Fast polarity switching was performed to extend the screening panel of pesticides covering positive and negative ESI.

Analysis was performed using the *Scheduled* MRM™ pro algorithm to reproducibly and accurately monitor hundreds of pesticides. In addition, 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-102 Targeted Identification and Quantitation of Pesticide Residues using Advanced MRM Scheduling on a Triple Quadrupole LC-MS/MS

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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 a variety of food matrices. Here we present a high-throughput routine LC-MS/MS method that combines screening with identification based on Multiple Reaction Monitoring (MRM) and full scan MS/MS data.

Fruit and vegetable samples from local supermarkets were extracted using a QuEChERS procedure and injected into LC-MS/MS after dilution to minimize possible matrix effects. LC separation was performed using Phenomenex Kinetex (50 x 2.1 mm) column and a gradient of water and methanol and ammonium formate buffer with a total run time of less than 20 min. Detection was performed on an SCIEX Triple Quad™ 3500 system using Electrospray Ionization.

Targeted pesticides were quantified and identified using the Scheduled MRM™ pro algorithm. This new algorithm allows setting of flexible detection windows for each target compound, dynamically extends the detection window if needed, and triggers qualifier MRM transitions when the quantifier is present: resulting in enhanced selectivity, sensitivity, accuracy, and reproducibility. The MRM ratio was used for pesticide identification and is automatically calculated in MultiQuant™ software.

The method provided sufficient sensitivity, accuracy and reproducibility to quantify and identify all targets at a concentration of 10μg/kg or below.

P-103   Application of High Resolution for Targeted Screening for Veterinary Drugs in Food

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Liquid Chromatography coupled to tandem Mass Spectrometry (LC-MS/MS) is a powerful analytical tool for the analysis of polar, semi-volatile, and thermally labile compounds of a wide molecular weight range, such as veterinary drugs, pesticides, mycotoxins and other food residues and contaminants. Mass analyzers based on triple quadrupole technology operated in Multiple Reaction Monitoring (MRM) mode deliver highly selective and sensitive quantitative results and are therefore well established for multi-target screening and quantitation. However, the use of triple quadrupole based mass analyzers limits the number of compound to quantify and identify. In addition there is an increasing demand for retrospective and non-target data analysis. High resolution and accurate mass instruments are capable of performing targeted and non-targeted screening in a single LC-MS/MS run.
Here, a generic procedure was used to extract residues and contaminants from food samples. Extracts were subsequently analyzed by LC-MS/MS using the SCIEX TripleTOF® 6600 system with *Ion*Drive Turbo V™ source operated in high resolution accurate mass MS-IDA-MS/MS mode.
Full scan MS and MS/MS data was explored to identify targeted compounds using extensive XIC lists. Analytes were identified with high confidence based on retention time matching, mass accuracy, isotopic pattern, and MS/MS library searching. The challenging data processing workflow was automated and allows easy result review and reporting in the latest vision of MasterView™ software.
In this study samples from a EU proficiency test were analyzed including tissue, liver and urine extracts.

P-104   Data Processing for High Resolution LC-MS/MS for Target Quantitation and General Unknown Screening

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There is an increasing demand for analytical techniques and methods combining targeted identification and quantitation with retrospective and non-target data analysis. High resolution and accurate mass instruments are capable of performing targeted and non-targeted screening in a single LC-MS/MS run.
A generic extraction procedure was used to extract residues and contaminants from food samples. Extracts were subsequently analyzed by LC-MS/MS using a new SCIEX high resolution system operated in high resolution accurate mass MS-IDA-MS/MS mode and SWATH™ MS/MS[ALL] mode.
Full scan MS and MS/MS data was explored to confidently identify and accurately quantify targeted chemicals based on retention time, accurate mass, isotope pattern and MS/MS library searching.
In addition, sample-control-comparison was successfully used to find unexpected contaminants. Identification was based on accurate mass MS and MS/MS information, including empirical formula finding, ChemSpider searching, and automatic MS/MS fragment ion interpretation. This challenging data processing workflow was automated and allows easy result review and reporting in the latest revision of MultiQuant™ and MasterView™ software.

P-105   Design of experiment versus "change and check": method optimization strategies for the determination of microcystins as markers of algae bloom contamination in surface waters by LC-ESI-TOFMS

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The advantages for mass spectrometry (MS) testing platforms have previously been reported for small molecule screening applications versus traditional ELISA methods at ppb levels. Of particular interest to this study is the emergence of MS as a means to monitor chemical markers of algae bloom contamination in surface waters. Time-of-flight (TOF) MS is ideal for this application, since researchers may not know the exact nature of contaminants for geographically isolated samples prohibiting targeted analysis approaches. Fast scanning TOFMS instruments offer identification by exact mass measurements, when paired with chromatographic separation tools (e.g. LC, UPLC) will

provide data that is more informing versus many orthogonal qualitative techniques. To maximize the performance of this instrument, multiple parameters must be optimized to allow the ion beam to move from the ionization source to the detector efficiently. This report details design of experiment (DOE) strategies to minimize heuristic efforts in this process. To verify the results, a "change and check" approach will also be reported to see if the final method parameters are the same or different and quantify the effect on method performance (LOD, LOQ, %RSD). A comparison of operational scan modes for the TOFMS will also be presented to detail the advantages in sensitivity with TrapPulse mode. Standards were prepared at Lake Superior State University (Sault Ste Marie, MI). Preliminary method development work was completed at the University of Central Florida prior to technology transfer to the PerkinElmer Center of Excellence (Oakbrook, IL).

P-106   Analysis of QuEChERS Extracts of a Variety of Foods for Pesticide Residues using Automated SPE Coupled to GC/MS/MS and LC/MS/MS

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QuEChERS extracts of foods and concentrated food ingredients can contain significant amounts of sugars, sterols, fatty acids, colors, and oils that can interfere with GC/MS/MS or LC/MS/MS analyses of pesticide residues. Effects commonly observed are suppression of pesticide ion formation, instrument fouling, and poor correlation of spiked sample responses with external or non-structurally related internal standards. We took great interest in the announcement (B. Morris et. al., NACRW – 2014) of new custom blends of SPE sorbents designed specifically to remove each of these interfering compound classes. As a result, we tested these sorbents in a variety of food measurements with focus on minimizing matrix effects. Also, we thoroughly evaluated this SPE approach for fully automated on-line SPE-GC/MS/MS and SPE-LC/MS/MS.

The new sorbent mixtures yielded highly effective clean-up of QuEChERS extracts. The degree of extract clean up achieved appears to significantly exceed the capability of dispersive SPE (most common approach for food analysis) in that it provided clear, nearly colorless extracts without loss of acidic or quaternary amine analytes. Furthermore, the pesticides Acephate,* Acibenzolai-s-methyl,* Aldicarb sulfone, Atrazine, Azoxystrobin , Carbaryl, Carbofuran,* Cyazofamid,* Dichlofluanid, Dimethoate, Ethoprophos,* Imidacloprid,* Linuron,* Methomyl,* Omethoate,* Phosmet,* Pymetrozine, Pyriproxyfen, Spinosyn A,* Spinosyn D,* Thifensulfuron-methyl,* Tricyclazole, Triflumizole* were all measured successfully by LC/MS/MS and those pesticides marked with an asterisk were most readily quantified using external, solvent only standards.   Using GC/MS/MS allowed the additional pesticides Lindane (g-BHC), Chlordane, Chlorothalonil, Chlorpropham, Chlorpyifos, Cypermethrin, o,p'-DDE, Deltamethrin, Diazinone, Endosulphan sulfate, Fenthion, Folpet, Phthalimide,* Pirimiphos methyl, Tetrahydrophthalimide, Toclofos methyl to be measured successfully in the extracts, but as is common, these GC based measurements relied more on the use of internal standards.

Testing this novel SPE clean-up, using miniaturized SPE cartridges, allowed column SPE to be performed with a robotic instrument autosampler (CTC PAL-xt), with the advantages of precisely controlled flow rates and elution volumes. The use of this approach allowed the total automation of the SPE-LC/MS/MS and SPE-GC/MS/MS measurements of QuEChERS extracts with cycle times of 15 min. We believe there is still significant room to decrease cycle time with further robot optimization.

P-107   A New Sorbent for Cleanup of Meat and Milk Extracts Prior to Multiresidue Veterinary Drug LC-MS Analysis

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Meat and milk samples are typically extracted with an acetonitrile based solvent for LC-MS determination of veterinary drug residues. Among the most significant co-extractable substances are fats and polar lipids, particularly phospholipids. For example, 1 mL of whole milk contains about 35 mg of fat and about 0.3 mg of phospholipids. A gram of pork muscle typically contains about 100 mg of fat and about 5 mg of phospholipids. Reversed-phase sorbents such as C18 are effective for removal of fat from the acetonitrile based extraction solvent, but are ineffective for removal of phospholipids. Excessive amounts of phospholipids can shorten LC column life and contribute to ion-suppression and contamination in the mass-spectrometer. Results indicate that this new sorbent is highly effective for removal of both phospholipids and fats from meat and milk extracts prior to LC-MS analysis. With the new sorbent, recoveries of veterinary drugs were similar to results obtained using C18 for cleanup, but phospholipid removal was greater than 80% better.

P-108 **Evaluation of a Modified Quechers Method for LC-MS Determination of Multiresidue Mycotoxins in Grain Flours**

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Recently a method was published describing a Quechers based method for determination of 14 mycotoxins in rice. In this poster an evaluation of that method is presented for determination of those mycotoxins in wheat flour, corn flour and brown rice flour. Excellent recovery was observed in all three matrices for most compounds including aflatoxins, fumonisins, ochratoxin, T2-Toxin and HT-2 toxin. Recovery of citrinin was good, but some instability of this compound was observed in the extracts of low level samples, particularly in the wheat flour extract. Results obtained using the original dispersive SPE cleanup procedure will be compared with results obtained using an alternative cleanup procedure.

P-109 **Atmospheric Pressure Ionization Coupled to Tandem Quadrupole Mass Spectrometry for the Analysis of Pyrethroids in Water Samples**

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Pyrethroid insecticides are used widely in agriculture to control crop damaging pests. They are often found at trace levels in nearby rivers due to runoff from fields that have been treated. These compounds present an analytical challenge under traditional EI GCMS due to heavily fragmented spectra being produced. Atmospheric Pressure Gas Chromatography (APGC) ionization utilizes a softer atmospheric pressure chemical ionization mechanism which produces a more intact precursor ion allowing easier identification and more selective MRM transitions. Data was acquired with APGC coupled to a tandem quadrupole mass spectrometer in the targeted analysis of pyrethroids in river water, with low energy precursor and high energy fragment full scan channels. Comparing the softer atmospheric pressure ionization technique with traditional EI GC ionization indicated the aforementioned conservation of the precursor ions. Ionization was performed with the use of water as a source reagent which produced [M+H]$^+$ ions. Following SPE, river water extracts were run along with a solvent standard calibration curve. Pre- and post-extraction spikes were compared to assess the recovery of the extraction method. A number of solvent QC samples were injected and showed that the reproducibility of the system for targeted analysis was suitable. Limits of detection for these compounds were found to be in the fg on-column range for solvent standards. The full scan low and high energy data was interrogated and additional non-targeted compounds were identified in the river water extracts.

P-110 **Screening for Perfluoroalkyl Substances (PFASs) in Wildlife and Environmental Samples Using a Highly Sensitive LC-QTof MS**

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Perfluoroalkyl substances (PFASs) encompass a range of fully fluorinated alkyl compounds, typically with an anionic end group. These compounds have been implemented in a range of consumer goods and industrial processes due to their hydro- and lipophobic properties. As a result of their widespread use and subsequent leaching from materials, they have been found in various environmental and biological samples. For monitoring and research purposes, sub-ppb detection of these compounds is often required, in particular for water analysis. Traditionally, this type of analysis has been performed using the selective MRM approach on a tandem quadrupole MS. However, the ability to look for other contaminants of concern post acquisition or matrix components such as co-extracted bile acids supports the use of QTof MS. In this work we demonstrate the low levels of detection (ppb and sub-ppb range) for known PFASs as well as their quantification in wildlife and water samples using exact mass fragment information for confirmation. For most of the analytes, a linear dynamic range of at least 2.5 orders of magnitude was observed. The ability to perform historical data review was also exploited in searching for emerging PFASs that were identified as compounds of interest post acquisition.

P-111   Demonstration of Collision Cross Section Value Conservation Across LC and GC Analyses

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Contaminant identification in food and environmental matrices using both GC and LC/MS techniques are widely implemented, although challenges with matrix effects, false detections and reproducibility of ion ratios exist. Here we demonstrate the application of ion mobility coupled to a quadrupole time-of-flight (QTof) MS in order to generate a robust and unique additional measurement for contaminant identification. The determination of the collision cross section (CCS) of an ion can be extrapolated from the observed drift time as the ion passes through the drift cell. In order to demonstrate the robust and precise nature of CCS values, pesticides were analyzed under both GC and LC conditions, and the CCS values compared.

Generic chromatographic methods for both the LC and GC separations were used to cover a wide range of chemical classes. Atmospheric pressure chemical ionization was used for GC-MS and electrospray ionization for LC-MS. An ion mobility enabled QTof MS was used for detection, operating in alternating low and high collision energy states in order to generate both precursor and fragment ions. From these results, it could be seen that CCS values represented a unique property of the ions generated that was well conserved, regardless of how the analytes were introduced (i.e. GC or LC) into the ion mobility MS system. These results support the use of CCS values, in addition to accurate precursor mass and fragment ions, for compound identification.   Several fruit and vegetable matrices were also assessed to demonstrate conservation of CCS in samples.

P-112   A Novel Strategy to Screen and Profile Steviol Glycosides of Natural Sweeteners in Food Using Microfluidic UPLC Ion Mobility Mass Spectrometry

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Stevia rebaudiana Bertoni is a perennial shrub of the Asteraceae (Composite) family native to regions of South America. It is of significant economic value due to the high content of natural sweeteners in its leaves. Currently, stevia plant or extracts are used as sweeteners in South/North America, Asia and some European countries. The Joint FAO/WHO Expert Committee on Food Additives (JECFA) established regulations for steviol glycosides demanding a purity level of at least 95% of the seven chemically defined steviol glycosides. Here we present a unique approach to screen food products for steviol glycosides, using microfluidic chromatography and ion mobility combined with high resolution mass spectrometry (IM-MS).

Analysis was performed using a prototype post column addition (PCA) microfluidic device in the negative ionisation mode. Collision cross sections (CCS), accurate mass, fragment ions and retention time were obtained to profile the steviol glycosides rebaudioside A to F, rubusodside, steviol, dulcoside A, steviolbioside and stevioside. CCS measurements were obtained for the marker standards at 100fg/µL, and this information was used to create a scientific library incorporating the expected steviol glycoside CCS values. Extract of chocolate spread was spiked with the standards, analysed and screened against the generated CCS library. When comparing the expected and measured collision cross sections, the CCS measurement errors were typically <0.4%. In addition, it has been possible to acquire mobility resolved fragmentation spectra, which are resolved from co-eluting components. Furthermore, the approach taken reduces the quantity of expensive commodities i.e. high purity standards and solvents.

P-113   Determination of Triphenylmethane Dyes and Their Metabolites in Shrimp Using a modified QuEChERS Extraction and LCMSMS

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Motivated by the various potential health benefits, global consumption of seafood continues to increase. In order to meet this demand, the practice of farming aquatic species has seen significant growth as certain areas of the world's fish stock become overexploited. One of the main challenges in the aquaculture industry is the control of infectious diseases.

Due to their efficacy and low cost, triphenylmethane (TPM) dyes including malachite green (MG), crystal violet (CV) and brilliant green (BG) have been implemented as antimicrobials to combat this problem. However, these compounds accumulate in fish and when this contaminated seafood is consumed by humans it poses a potential health risk. As a result, these dyes have been banned in many countries for use in seafood intended for consumption by humans. Despite which, TPM dyes are still being used in fish farming around the world. Sensitive and selective methods are needed to ensure the safety of seafood products.

A modified QuEChERs technique was employed for preparation of shrimp. An LC/MS/MS method for simultaneously analyzing MG, LMG, CV, LCV and BG was employed. Solvent and matrix matched calibration standards ranged from 0.05-40 ppb. Excellent linearity was achieved for all compounds with all r^2 values > 0.998. The limits detection were ten to twenty times lower than the FDA recommended maximum level of 1 µg/kg. Average recovery for LCV with IS correction was 104% and for LMG it was 106%. The average recoveries for MG, CV and BG without IS correction were 33%, 83% and 54% respectively.

P-114   Facile Identification of Potential Pesticide Violations using Non Targeted Data Acquisition in Combination with an Integrated Scientific Information System

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Data obtained from non targeted, data independent acquisition on a high resolution mass spectrometer can be used to screen for a theoretical unlimited number of target compounds. Information rich datasets collected using UPLC/MS^E provide accurate mass measurements for both precursor and fragment ion information in a single injection. Using the same acquisition and processing parameters, within an integrated scientific information system, it is also possible to screen for compounds of interest not present in the target list. Here we demonstrate the use of binary compare and halogen match tools to isolate masses of interest from food matrices. Facile identification of significant compounds was performed using a novel batch elucidation tool where elemental composition is performed on each mass of interest. Proposed chemical formulae are submitted to Chemspider and resulting structures are returned as mol files. These structures are automatically subjected to *in silico* fragmentation using chemically intelligent bond breakages of the parent molecule and then used to identify any possible accurate mass fragments in the high energy channel. Ranking results by number of high energy fragments matched or by citations number, within Chemspider, allows the user to immediately focus on the identified components.

P-115   A Single LC-MS/MS Method for Screening, Identification and Quantification of over 400 Pesticides in Complex Matrix without Compromising Data Quality

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More than 500 compounds are routinely used for crop protection across the globe. With increasing global trade there is a requirement for rapid multi-residue screening and quantification methods to efficiently determine residue violations and protect consumers. Effective multi-residue methods rely on management of the acquisition of a large number of MRM transitions. Setting up overlapping MRM windows based around the retention time of each analyte ensures that no time is wasted acquiring other transitions for compounds that have yet to elute. This optimises the time spent acquiring data to maximize sensitivity whilst ensuring sufficient number of data points across peaks to give good precision. One of the objectives of this method was to implement relatively wide windows, without any loss in performance, removing the need to make regular checks on retention time drift and avoiding the need to make adjustments to the acquisition method before each analysis.

A single fast method for screening, identification and quantification of more than 425 pesticides was developed. All pesticides were separated on reverse phased column within 12 minutes. Two MRM transitions for each pesticide were monitored using both ESI positive and negative modes (deploying polarity switching). The excellent performance of the method has been demonstrated at very low concentrations in chilli powder, which is a very complex matrix. Standard curves in solvent and matrix, the ease of use of instrument and software features to calculate the incurred residues in chilli sample and data showing robustness after a large number of injections will be presented.

P-116   A Simple, Reliable and Fast LC-MS/MS Method for Determination and Quantification of Phthalates in Distilled Beverages

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Phthalates, esters of phthalic acid, are often used as plasticizers for polymers such as polyvinylchloride. They are widely applicable in various products including personal care goods, cosmetics, paints, coatings and food packaging. Phthalates have been found to leach readily into the environment and food as they are not chemically bound to plastics. As such, they are known to be ubiquitously present in our environment. Phthalates have been reported to show a variety of toxic effects related to reproduction in animal studies, which has resulted in these compounds being considered as endocrine disruptors. Screening food and beverages for phthalates contamination is required by many legislative bodies.
Seven phthalates were separated on a reversed-phase column within 11 minutes using Ultra Performance Liquid Chromatography coupled to a tandem quadrupole mass spectrometer. The dilute and shoot approach was used for sample preparation and subsequent analysis.   In order to assess the method's applicability, various brands of distilled spirits were tested. Repeated injections of the samples were made to evaluate method robustness over a number of days. Limits of detection (LOD) and quantification (LOQ) of seven key phthalates (DEHP, BBP, DBP, DNOP, DEP, DMP and DINP) in the sample will be presented.

P-117   Determination of the Metabolites of Nitrofuran Antibiotics in a Range of Animal Tissues and Associated Products by Liquid Chromatography-Tandem Quadrupole Mass Spectrometry

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Nitrofurans are a group of broad spectrum antibiotics employed as feed additives for prophylactic and therapeutic treatment of bacterial and protozoan infections. Due to health concerns, nitrofurans were banned from use in food animal production in many countries including the European Union (EU). Countries with products intended for the EU are bound by the same regulations as locally produced food therefore food imported into the EU should be free of nitrofurans. Over the past twelve years there have been frequent findings of residues in poultry and aquaculture products imported to EU countries leading to product recalls, border rejections and de-listed suppliers. These violations have resulted in an increase in pre-harvest (PHT) and pre-export (PET) testing and analysis of imports at border control. The European Commission prescribed analytical performance limits and criteria to be met to report a sample as non-compliant and a Minimum Required Performance Limit (MRPL) of 1 µg/kg for furazolidone, furaltadone, nitrofurazone and nitrofurantoin, measured as their respective tissue-bound metabolites. Laboratories must demonstrate that their calculated analytical performance limits (Detection Capability CCb and Decision Limit CCa) are at or below the MRPL. Although enforcement action is only taken where a residue exceeds the MRPL, non-complaint samples below the MRPL must still be monitored. Meeting these requirements requires the continued development of highly sensitive and specific analytical methodology based upon liquid chromatography-tandem quadrupole mass spectrometry (LC- MS/MS). Examples of performance are given for the analysis of nitrofurans in prawn, fish, honey, egg, poultry muscle, bovine kidney and feed.

P-118   Rapid, Direct Technique for the Discrimination of Meat Tissues Originating from Different Animal Species for Food Authenticity

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Fraudulent adulteration of food products with meat from undeclared species is a problem on a global scale, as exemplified by the European horse meat scandal in 2013. Introduction of undeclared meat into the food chain is a significant problem for consumers from an ethical or religious viewpoint and undermines confidence in food chain traceability and safety. Laboratory methods used for determination of meat speciation and adulteration (*e.g.* ELISA, PCR or proteomics) are time consuming and costly. Rapid Evaporative Ionisation Mass Spectrometry (REIMS) is an emerging technique that allows rapid characterization of biological tissues. Here we applied the same technology to provide direct

analysis of meat for the identification of species and the level of adulteration in near real time.

Meat was sampled using an electrosurgical device. The "smoke" aerosol generated was transferred to the mass spectrometer by a Venturi air jet pump-based ion transfer apparatus mounted in the orthogonal position relative to the atmospheric interface of a time of flight mass spectrometer. Although spectra acquired from horse and beef meat look similar, the profile of these 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 a PCA-LDA model. The models were verified with cross-validation and independent test sets. Preliminary data shows successful separation of pure horse and beef meat and the limit of detection for horse in beef patties was found to be 1% in compliance with UK labeling legislation.

P-119   Screening for Melamine, Cyanuric Acid and Dicyandiamine in Powdered Milk and Infant Formula using Liquid Chromatography- Mass Detection

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Melamine and cyanuric acid are low mass, nitrogen-rich compounds which have been linked to protein adulteration in various foodstuffs in the past. While not particularly toxic individually, in combination they can sometimes form an adduct compound through hydrogen bonding, melamine cyanurate, which can produce sharp crystals causing internal organ failure and possible death. A similar compound, dicyandiamide, which is used to minimize the environmental impact of grazing livestock has also been detected in small amounts in dairy products in New Zealand. Permitted limits on melamine are 1 mg/kg in infant formula and 2.5 mg/kg in other foods and animal feed. These values are based on the TDI (tolerable daily intake) of melamine and its analogues of 0.64 mg/kg body weight. Recently a more stringent TDI of melamine and its analogues of 0.2 mg/kg body weight was established. For DCD, the European Food Safety Authority has established a TDI of 1 mg/kg body weight.

As these compounds are quite polar, reverse phase methods do not work well. Current methods employ HILIC chemistry or ion pair mechanisms. Here, a rapid screening method for melamine, cyanuric acid and dicyandiamide in infant formula is presented. Recoveries for the three analytes studied were in the range of 75-123% for the variety of samples studied. Baseline separation was achieved within 3 minutes for all analytes and detection was conducted on a single quadrupole mass detector. Excellent linearity (>0.995) and repeatability (%RSD <10, n=7) was achieved for all three analytes and will be discussed.

P-120   A Rapid Screening Assay for the Simultaneous Detection of Antimicrobial Agents in Bovine Milk by Liquid Chromatography Coupled with an Accessible Mass Detector

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An important aspect relating to the use of veterinary medicines in cattle is the presence of residues in milk. Antimicrobials constitute the largest class of compounds administered to livestock globally. This widespread use together with stringent food safety legislation necessitates the availability of rapid and sensitive analytical techniques for residue detection. Cost-effective, robust and broad-scope platforms, which can be easily implemented in routine control laboratories are of importance. Other considerations are the flexibility of analytical scope and the extent of compliance with internationally recognised validation criteria.

The screening analysis of antimicrobial residues, as required by European Union (EU) Regulation 2002/657/EC will be discussed on a compact single quadrupole mass detector. A simple dispersive solid phase extraction (d-SPE) procedure provided effective and simple sample clean up, where average recoveries were > 90 % for all multi class analytes. Liquid chromatography separation, coupled with an accessible single quadrupole mass detector provided robust analysis and high sample throughout, where the screening target concentrations (STC) were less than the EU MRLs for each analyte. The method was found to be fit for purpose, thus allowing for reliable and rapid screening of commonly administered multi residue antibiotics below EU regulatory limits in bovine milk.