51st Annual

NACRW

NORTH AMERICAN CHEMICAL RESIDUE WORKSHOP

formerly Florida Pesticide Residue Workshop

TradeWinds Island Grand Resort
St. Pete Beach, Florida
July 20-23, 2014

"Bringing Scientists Together to Develop and Validate Better Methodologies"

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FUTURE MEETING DATES

2015 July 19 - 22

Meeting will be held at the TradeWinds Island Grand Resort St. Pete Beach, Florida

NACRW
NORTH AMERICAN CHEMICAL RESIDUE WORKSHOP
formerly Florida Pesticide Residue Workshop

July 20, 2014

Dear Attendees, Exhibitors, and Guests,

Welcome everyone to the 2014 North American Chemical Residue Workshop! We offer an especially warm welcome to our new guests and international friends. And special thanks to our volunteers, exhibitors and sponsors for their generous support of the 51st Workshop.

The 2014 NACRW Organizing and Program Committees have worked together throughout the year to provide interesting scientific sessions, many opportunities that will foster discussion and promote our collective knowledge in chemical residue and contaminant analysis. (And have a little fun along the way!)

For our Short Course, we are excited to have Professor Nicholas Snow, Ph.D., Chair of the Department of Chemistry and Biochemistry Seton Hall University. Professor Snow's interests focus on extending the boundaries of existing analytical techniques to lower detection limits and widen the range of application. This year's short course titled: *"Fundamentals of Gas and Liquid Chromatography for Residue Analysis"* will place emphasis upon gas chromatography and include discussion of the setup and operation of instrumentation with a focus on sampling, inlets and detectors.

Our welcoming reception on Sunday evening will kick-off the 51st NACRW. This will take place on site in the Pavilion exhibit area of the hotel. This social time with our exhibitors is always a great way to begin our week together. Then on Monday night we will be enjoying an evening dinner cruise with drinks and dancing on the tropical waters of *Boca Ciega Bay.* We hope you all will enjoy these and other relaxed events. Special thanks to our many sponsors for helping us keep the costs to a minimum!

The ever popular Mass Spec Forum will occur on Wednesday, following the Closing / Poster Awards. The session is a moderated group discussion focusing on issues associated with targeted and non-targeted methods. Bring your really tough questions to this highly interactive meeting as Walter Hammack and Mark Crosswhite have yet to be stumped!

Many are saying the 2014 Program Committee general theme, "Science without Borders", looks to be one of the best three day programs ever! Sessions Include: *Veterinary Drug Residue Analysis, New Approaches to Ensure Quality, Pesticide and Bees: Analysis Tools and Toxicological Effects, High Throughput Sample Prep and Analysis Methods, Perfluorinated Compounds* and two sessions dedicated to highly interesting general topics.

Nearly 150 posters and oral presentations will be offered this year. Please take time to engage the authors, ask questions and cast your all-important vote for the best poster. The Journal of Agricultural and Food Chemistry is again offering to publish a special edition affiliated with NACRW. Should you have a manuscript to submit for publication, please inform Perry Martos or Paul Yang.

Vendor workshops start on Sunday evening and occur each day of the workshop. Check the schedule included and look for signup sheets near the registration desk for times and locations. Please make sure to visit the Exhibition for one-on-one discussions with vendors. And for those staying through Thursday, there are a few vendors holding users meetings on morning of July 24th.

The NACRW Organizing Committee Meeting takes place on Tuesday from 5:45 to 6:25 pm. NACRW is a volunteer organization; please consider participating in the Organizing and/or Program Committee. You are encouraged to become actively involved in this outstanding meeting. NACRW is sponsored by FLAG Works, Inc., a not-for-profit organization.

We would like to thank the 2014 Organizing Committee, Program Committee and all of the volunteers for arranging and participating in this great meeting. Finally, a very special thank you to Mrs. Teri Besse who makes our meeting work…
Please Enjoy!

Brad Barrett, Michael Telepchak, Julie Kowalski, Gale Hagood and Shirley Elliott, (2014 Organizing Committee)
Perry Martos, Paul Yang, (2014 Program Committee Co-Chairs) and the Program Committee members

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ChromaBLOGraphy is where Restek's renowned experts go to share their thoughts on current trends along with best practices and troubleshooting tips. Better yet, you have the opportunity to weigh in yourself.

Here's a look at some of our latest posts:

- *Are Fatty Acids Overwhelming Your QuEChERS dSPE PSA Cleanup and Causing Issues in Your GC Analysis?*
- *Shortcut: AOAC QuEChERS Protocol*
- *Believe to Achieve: Evaluating the Accuracy of the EZGC™ Chromatogram Modeler*
- *Maybe a Rose is a Rose is a Rose, but a "5" is not a "5", When it Comes to Pesticide Analysis….*
- *Resurrecting an Old Reversed-Phase LC Column — Should I, Could I, How Would I?*
- *Chemstation 101 – Restoring a Previous Tune File*
- *I'll Stop the Autosampler and Melt the LVI With You….*
- *Troubleshooting HPLC — Fronting Peaks*
- *Analyzing Residual Solvents in Cannabis Concentrates: A Sticky Situation*

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East
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Fitness Center
Portico
Allamanda Bldg.
Jacaranda Bldg.
Garden Courtyard
Banyan Breezeway
POOL & HOTTUB AREA
Hibiscus
Hibiscus
Pet Play Zone
Sharktooth Tavern
Parking
VENDOR SEMINARS
Palm Court
Island Ballroom
Grand Palm Colonnade CONFERENCE CENTER
South Parking
Jacaranda Hall
Paddleboat Landings
Pirate Island
POOL AREA
TAZ Activities
Tennis Courts
The Pavilion
Parking
Cypress Villa
Parking
North Parking
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REGISTRATION DESK AND MEETING ROOM
Magnolia Offices
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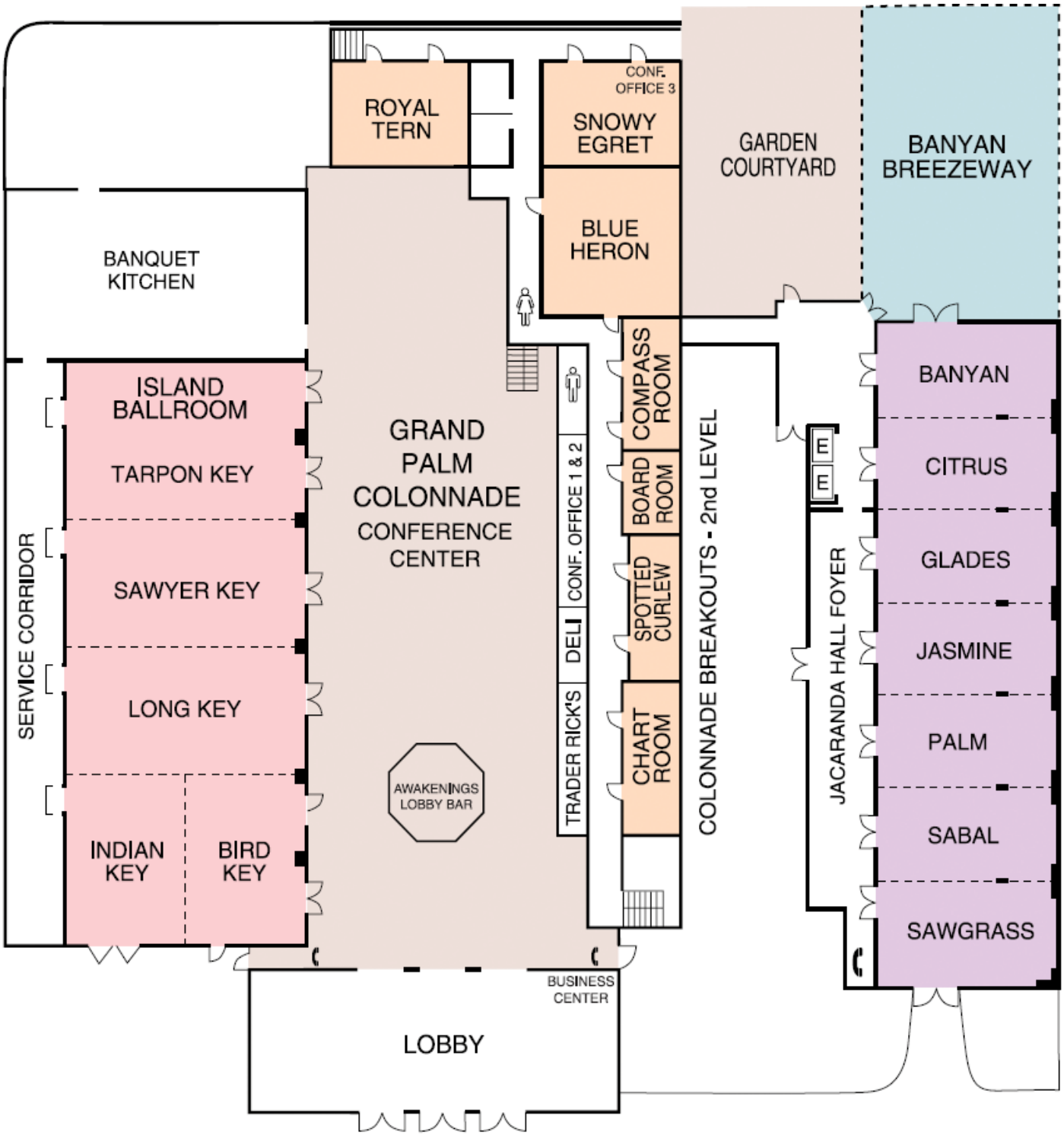

Technical Sessions: Long Key, Bird Key and Indian Key Ballrooms
Exhibits, Posters, Reception: The Pavilion
Vendor Seminars: Tarpon Key

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MEETING AT A GLANCE

Saturday, July 19, 2014		
8:30 am - 4:00 pm	Short Course – Dr. Nicholas Snow	Glades
Sunday, July 20, 2014		
8:30 am - 4:00 pm	Short Course – Dr. Nicholas Snow	Glades
1:00 – 5:00 pm	Exhibitor Setup	Pavilion
2:00 – 6:00 pm	Registration	Grand Palm Colonnade
6:15 – 7:15 pm	Restek Vendor Seminar	Tarpon Key
7:30 – 8:30 pm	Welcome Reception	Pavilion
Monday, July 21, 2014		
7:30 am – 5:00 pm	Registration	Grand Palm Colonnade
7:00 – 10:00 am	Poster Board Set Up	Pavilion
7:45 – 8:15 am	Early Morning Coffee	Grand Palm Colonnade
7:15 – 8:15 am	Waters Corporation Vendor Seminar	Tarpon Key
8:30 – 10:45 am	**Science without Borders -Opening Session**	Ballrooms
11:00 – noon	Exhibition and Poster Opening	Pavilion
11:05 – 11:50 am	Poster Session A1 (authors for odd #s)	Pavilion
12:10 – 1:10 pm	Thermo Scientific Vendor Seminar	Tarpon Key
1:15 – 2:55 pm	**Veterinary Drug Residues**	Ballrooms
3:00 – 3:50 pm	BREAK (Exhibition & Posters)	Pavilion
3:05 – 3:50 pm	Poster Session B1 (authors for even #s)	Pavilion
4:00 – 5:15 pm	**New Approaches to Ensure Quality**	Ballrooms
6:00 pm	Dinner Cruise (Bus Departure)	Depart from Hotel Lobby
Tuesday July 22, 2014		
8:00 am – 5:00 pm	Registration	Grand Palm Colonnade
7:45 am – 5:00 pm	Exhibition & Posters	Pavilion
7:45 – 8:15 am	Early Morning Coffee	Pavilion
7:15 – 8:15 am	AB SCIEX Vendor Seminar	Tarpon Key
8:30 – 10:35 am	**Pesticides and Bees: Analysis Tools and Toxicological Effects**	Ballrooms
10:45 – noon	BREAK (Exhibition & Posters)	Pavilion
11:00 – 11:45 am	Poster Session A2 (authors for odd #s)	Pavilion
12:10 – 1:10 pm	Agilent Technologies Vendor Seminar	Tarpon Key
1:15 – 2:55 pm	**Updates from State and Government Laboratories**	Ballrooms
3:00 – 3:55 pm	BREAK (Exhibition & Posters)	Pavilion
3:05 – 3:50 pm	Poster Session B2 (authors for even #s)	Pavilion
4:00 – 5:40 pm	**High Throughput Preparation and Analysis Methods**	Ballrooms
5:45 – 6:25 pm	**Organizing Committee Meeting**	Ballrooms
6:30 – 7:30 pm	Bruker Daltonics Vendor Seminar	Tarpon Key
7:45 pm	Beach Volleyball Game	On the Beach
Wednesday, July 23, 2014		
6:15 am	Beach Walk/Run	On the Beach
8:00 am – noon	Registration	Grand Palm Colonnade
7:45 am – noon	Exhibition & Posters	Pavilion
7:45 – 8:15 am	Early Morning Coffee	Pavilion
7:15 – 8:15 am	Thomson Instrument Company Vendor Seminar	Tarpon Key
8:30 – 10:35 am	**Perfluorinated Compounds and Emerging Contaminants**	Ballrooms
10:45 – noon	BREAK (Exhibition & Posters)	Pavilion
12:10 – 1:10 pm	Phenomenex Vendor Seminar	Tarpon Key
1:15 – 2:55 pm	**General Topics I**	Ballrooms
3:00 – 3:15 pm	BREAK	Grand Palm Colonnade
3:15 – 4:55 pm	**General Topics II**	Ballrooms
4:55 – 5:10 pm	**Closing (Poster Award)**	Ballrooms
5:20 – 6:30 pm	**Informal Forum on Mass Spectrometry**	Ballrooms
Thursday, July 24, 2014	User Meetings	
7:30 – 9:30 am	AB SCIEX	Banyan
10:30 – 12:30 pm	Agilent	Citrus
10:30 – 12:30 pm	Thermo Scientific	Glades

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GENERAL INFORMATION

Registration

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

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 authors must be at their posters from 11:05 am – 11:45 am on Monday and 11:00 - 11:45 am on Tuesday
Poster Session B authors must be at their posters from 3:05 pm – 3:50 pm on Monday and 3:05 – 3:50 pm 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, we will have a new award this year, the UCT SPE Award. **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 H'orderves and open bar 7:30 - 8:30 pm
Tuesday - 7:45 am – 5:00 pm
Wednesday - 7:45 am – noon

Coffee and Breaks

Coffee will be available 7:45 - 8:15 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 24th, 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 NACRW-FPRW in 2014. Please inform Perry Martos (pmartos@uoguelph.ca) or Paul Yang (paul.yang@ontario.ca) 2014 Program Co-Chairs, by September 5, 2014 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 October 31, 2014.

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 you a reprint.

Meeting Website

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

Meeting Evaluations

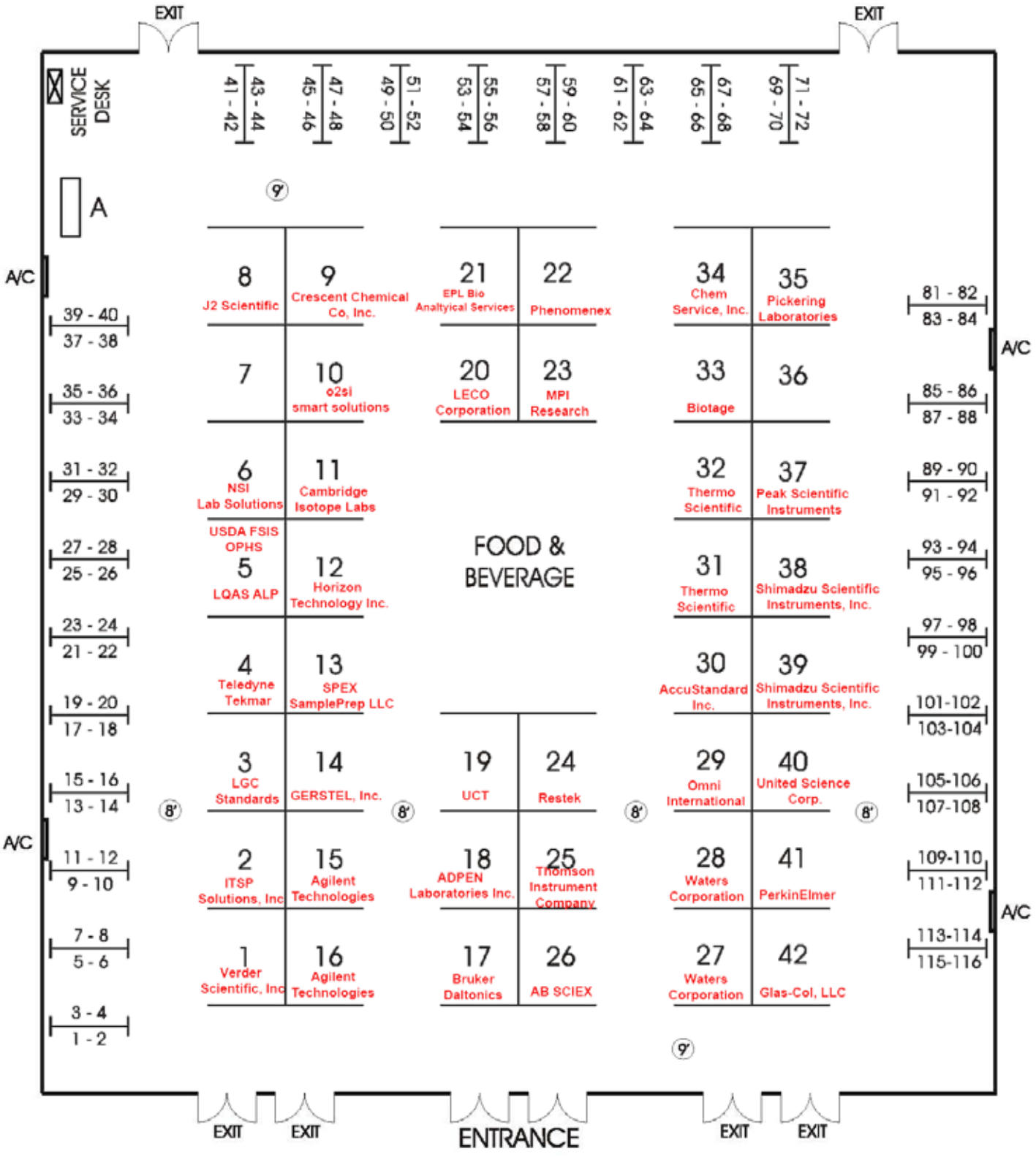
Look for on-line conference evaluations this year! Evaluations will be emailed to you daily, so please take a few moments each day to fill them out.

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

FUTURE MEETING DATES
2015 July 19 - 22
All Meetings will be held at TradeWinds Island Grand Resort, St. Pete Beach, Florida

Exhibits and Poster Sessions

Location: Pavilion



Exhibitors

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www.ADPEN.com

Agilent Technologies
Booth #15 and 16
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SHORT COURSE

Saturday, July 19, 2014 8:30 am to 4:00 pm
Sunday, July 20, 2014 8:30 am to 4:00 pm
Location: Glades

Fundamentals of Gas and Liquid Chromatography for Residue Analysis

Instructor: Nicholas Snow, Ph.D.
Professor and Chair
Department of Chemistry and Biochemistry
Seton Hall University

This two day course will cover the fundamentals of gas and liquid chromatography applied to residue analysis. The first day will focus on gas chromatography and include discussion of the setup and operation of instrumentation with a focus on sampling, inlets and detectors. The use of newer technologies including GCxGC, MS-MS and exact mass detection will be emphasized. The second day will focus on liquid chromatography with emphasis on columns, method development and detection, including LC-MS. Both days will conclude with a troubleshooting workshop and open discussion. Participants are encouraged to bring questions and problem examples from their own work for discussion. PRE-REGISTRATION IS REQUIRED.

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VENDOR SEMINARS

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

V-1 Sunday Evening, July 20, 2014, 6:15 p.m. to 7:15 p.m. RESTEK

Location: Tarpon Key

An Introduction to EZGC™ Method Development Tools: Method Translator and Flow Calculator

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

The EZGC™ Method Translator and Flow Calculator are tools for gas chromatography method development. Generally, the goal of Method Translation is to allow alteration of GC column format, carrier gas, flow, etc., while keeping peak elution order (NOT retention times) the same. Note that Method Translation assumes that the GC stationary phase type is always the same for Original and Translation methods. The Flow Calculator quickly provides information for average velocity, holdup time, inlet pressure, etc., based on GC column flow, and also calculates valve time for splitless injections based on liner volume and inlet temperature and flow conditions.

This presentation will demonstrate practical uses for the Method Translator, including:

Increasing speed of analysis through column length decrease and/or inside diameter decrease and/or switching to a faster carrier gas (e.g. going from helium to hydrogen).

Updating the oven temperature program through Translation after column trimming for maintenance so peak elution orders do not change.

Improving Original methods in separation and/or speed of analysis by solving for Efficiency or Speed in Translation.

All seminar attendees will be given a copy of the EZGC™ Method Translator and Flow Calculator on a USB drive. Attendees can answer a fun quiz and win excellent prizes!

V-2 Monday, July 21, 2014, 7:15 to 8:15 am WATERS CORPORATION

Location: Tarpon Key

Characterization of Mixed-Halogen Dioxins and Furans in Fire Debris using GCXGC-TOFMS and APGC-TQS

Frank L. Dorman, Ph.D., Associate Professor of Biochemistry and Molecular Biology
The Pennsylvania State University, 107 Whitmore Laboratories, University Park, PA, fld3@psu.edu

Large scale fires such as the Plastimet fire (Ontario) and The World Trade Centers (New York City) have raised concern about long term exposure of firefighters to the combustion byproducts of brominated flame retardants (BFRs). The wide spread use of brominated flame retardants in common consumer products has extended the concern of health implications to long term exposure of first responders to fire debris. Studies on the contents of fly ash from municipal waste incinerators have documented that mixed halogenated dioxins and furans (PXDD/Fs) are created from the combustion of flame retarded materials.

This study focuses on the investigation of the role mixed halogenated planar compounds, generated during the combustion of BFR-containing products; play in the toxicity experienced in first responders. The emphasis of the project is placed on the generation of polyhalogenated dibenzo-p-dioxins and dibenzofurans in residential or commercial fires situations. An Atomspheric Pressure Gas Chromatography (APGC) coupled to a Xevo TQ-S-MS method for analysis of the fire debris samples for mixed halogen dioxins and furans is in development. Considering that there were no dioxin congeners identified in GCxGC-TOFMS analysis of the fire debris samples, it was postulated that the dioxin congeners were present, but at levels below the limit of detection of the TOF-MS. Therefore, the sensitivity of the APGC-TQ-S is beneficial for identifying these compounds.

V-3 Monday, July 21, 2014, 12:10 to 1:10 pm THERMO SCIENTIFIC

Location: Tarpon Key

Simplifying Contaminants Analysis – The Very Latest Developments in GC and LC MS Technology for Routine Screening and Quantitation of Contaminants in Complex Samples

Walter Hammack, Florida Department of Agriculture and Consumer Services
Salvador Lopez, Institute of Food Safety and Health

GC-MS and LC-MS are well established as the “routine workhorse” in many laboratories that are responsible for measuring pesticides and other contaminants in a diverse range of samples. These technologies offer a high degree of sensitivity through selectivity, and as a result enable opportunities for powerful, high efficiency methods. These methods are often high capacity, measuring hundreds of compounds with generic sample preparation approaches upstream of the instrumentation that deliver highly complex extracts and difficult matrix challenges. For optimum productivity, the systems employed need to be smart, robust, powerful and easy to use in the routine environment. They also need to deliver as much information as possible to help us understand our samples and methodologies in detail. In this seminar, we will focus on the very

latest developments in GC and LC mass spectrometry systems as well as complementary technologies that specifically address these needs. Hear Walter Hammack of the Florida Department of Agriculture and other industry leaders speak about their results and latest developments.

V-4 Tuesday, July 22, 2014, 7:15 to 8:15 am AB SCIEX
Location: Tarpon Key

What's In Your Food? See the Tools To Accurately Find & Identify Residues and Contaminants - All the Time, Every Time

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

Food testing can be a challenging and complex job. From sample preparation (so many different matrices!) to residue detection (so many different compounds from pesticides and mycotoxins, and not to mention the mysterious unknowns!), going from the raw sample to the final result of "What is in this food sample?" is no trivial task.

Luckily, a number of analytical tools and workflows are available to ease the pain and help you to answer the question above, quickly and efficiently, but also with the confidence that you arrived at the right result, every time.

In this workshop, we will describe LC-MS/MS technology and software tools that will make your food testing workflows better than ever. Learn how you can:

- Reduce the risk of reporting a false positive result
- Lower the likelihood of missing a result (fewer false negatives)
- Eliminate matrix interferences once and for all
- Screen large sample sets for hundreds of compounds in 5 mouse clicks
- Quantitate large data sets (100s of compounds in 100s of samples) in less than 5 minutes
- Easily and effectively identify unknown, or non-targeted mystery peaks (TRUE unknown screening)

V-5 Tuesday, July 22, 2014, 12:10 to 1:10 pm AGILENT TECHNOLOGIES
Location: Tarpon Key

Tools for Identifying Pesticide Metabolites in Fruits and Vegetables Using Accurate Mass LC/Q-TOF MS

Jerry Zweigenbaum, Ph. D., Agilent Technologies

This presentation will focus on the tools available in MassHunter Software that can be used to identify metabolites of pesticides in plant material. In the allotted time, a workflow will be described using different acquisition modes. Data processing will include the use of metabolite ID (briefly), using a diagnostic ion with all ions and AutoMSMS, using AutoMSMS and neutral loss, a molecular formula generator filter, MassProfiler, and MassProfiler Professional with Profinder.

The same tools can be used for the identification of metabolites of other classes of compounds and in other types of samples (e.g. urine or blood).

My QuEChERS Extraction is going on a Diet!

Joan Stevens, Ph.D., Agilent Technologies

The benefits of QuEChERS are well documented making this technique very popular and easily executed in any laboratory using common equipment such as pipettes, balance, vortexer and centrifuge. Because of the benefits QuEChERS provides as a sample preparation technique it is rapidly expanding to other matrices and compound classes. It is true that QuEChERS is a simple and easy technique, but the QuEChERS procedure does not entirely remove the matrix, which can negatively affect your analysis and instrumentation. Lipids, for instance, are a very problematic component remaining in the QuEChERS extraction especially with high lipid content samples like avocadoes and oils. Dispersive-SPE and cartridge SPE containing C18, hydrophobic sorbent, are used to remove some of the lipids from the extract but many times a large percentage of lipids remains. In this session we will present alternative approaches for increasing lipid removal from QuEChERS extract.

V-6 Tuesday, July 22, 2014, 6:30 to 7:30 pm BRUKER DALTONICS

Location: Tarpon Key

Mass Spectrometric Strategies for Accurate Screening and Quantitation of Chemical Residues

Joe Anacleto, Bruker Daltonics, 40 Manning Road, Billerica, MA 01821, 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. In addition, inductively coupled plasma mass spectrometry is an ideal tool for rapid, low level detection of inorganic contaminants within various products. This presentation will provide an overview of Bruker's lab-based chromatographic and mass spectrometric systems and how they provide market leading performance, ruggedness and ease-of-use when used with our innovative software solutions. Real world examples will include the rapid screening of pesticides in food with an ultra-high resolution LC-QTOF system, the targeted quantification of residues in food and water with both GC-TripleQuad and LC-TripleQuad systems, and the low level detection and speciation of inorganic contaminants in food.

V-7 Wednesday, July 23, 2014, 7:15 to 8:15 am THOMSON INSTRUMENT COMPANY

Location: Tarpon Key

New Sample Preparation Methodology to enable Higher Recovery, and minimize loss of difficult Analytes in Pesticide and Fungicide Panels by LC/MS or GC/MS

Lisa Wanders, Sam Ellis, and Joe Machamer, Thomson Instrument Company, Oceanside, CA

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 (which need to be concentrated). Several studies comparing these techniques with eXtreme filter vials (patented) for contaminant analysis were conducted in orange juice, soil, milk, shellfish and water analysis.

V-8 Wednesday, July 23, 2014, 12:10 to 1:10 pm PHENOMENEX

Location: Tarpon Key

A New Perspective in Pesticides Analysis: You Now Have a Choice – GC/LC/Sample Prep

Kristen Parnell, Phenomenex, 411 Madrid Avenue, Torrance, CA 90501, USA; KristenP@phenomenex.com

Historically, pesticide testing has been driven by regulatory requirements in sample preparation, GC and LC techniques. Traditional regulatory approaches in the testing of pesticides, herbicides, PCBs, and related compounds have been sufficient. However, recent technological and intellectual advancements in chromatography now provide more choices and options to improve your analytical success. This presentation will provide an overview and a perspective on different options that are often overlooked for pesticides testing.

MEETING PROGRAM

Saturday, July 19, 2014

8:30 am-4:00 pm **Short Course: Part 1** **Glades**
Fundamentals of Gas and Liquid Chromatography for Residue Analysis
Prof. Nicholas Snow, Seton Hall University

Sunday, July 20, 2014

8:30 am-4:00 pm **Short Course: Part II** **Glades**
Fundamentals of Gas and Liquid Chromatography for Residue Analysis
Prof. Nicholas Snow, Seton Hall University

1:00 – 5:00 pm Exhibitor Setup Pavilion
2:00 – 6:00 pm Registration Grand Palm Colonnade

6:15 – 7:15 pm **Restek Evening Seminar** **Tarpon Key**
V-1 **An Introduction to EZGC™ Method Development Tools: Method Translator and Flow Calculator**
Jonathan Keim, Jack Cochran, Julie Kowalski, Michelle Misselwitz, Chris Nelson
Restek Corporation, Bellefonte, PA, USA

7:30 – 8:30 pm Welcome Reception Pavilion

Monday, July 21, 2014

7:30 am – 5:00 pm Registration Grand Palm Colonnade
7:00 – 10:00 am Poster Board Set Up Pavilion
11:00 am Exhibition & Posters Pavilion
7:45 – 8:15 am Early Morning Coffee Grand Palm Colonnade

7:15 – 8:15 am **Waters Breakfast Seminar** **Tarpon Key**
V-2 **Characterization of Mixed-Halogen Dioxins and Furans in Fire Debris using GCXGC-TOFMS and APGC-TQS**
Frank L. Dorman, Ph.D., Associate Professor of Biochemistry and Molecular Biology
The Pennsylvania State University, University Park, PA, USA

8:30 – 10:50 am **Opening Oral Session:** **Ballrooms**
Science without Borders
Moderator: Jo Marie Cook (FDACS, FL, USA)

8:30 – 8:50 am **FLAG Works/President, Organizing Committee**

8:50 – 9:15 am **Jo Marie Cook** – Florida Dept. of Agriculture and Consumer Services, FL, USA
O-1 **Science without Borders**

9:20 – 9:45 am **Jack Kay** – Veterinary Medicines Directorate, UK
O-2 **Can One Regulation or Analytical Method Fit All Internationally?**

9:50 – 10:15 am **Anton Kaufmann** – Official Food Control Authority of the Canton of Zurich, Switzerland
O-3 **Multi-residue Methods –Complex Performance Criteria Considerations**

10:20 – 10:45 am **Louis Bluhm** – USDA FSIS OPHS, GA, USA
O-4 **How USDA FSIS Assesses Quality Assurance in Chemistry Methods**

11:00 – noon **BREAK, Exhibition Opening & Posters** **Pavilion**
11:05 – 11:50 am **Poster Session A1** **Pavilion**
Authors of odd poster numbers present

12:10 – 1:10 pm **Thermo Scientific Lunch Seminar** **Tarpon Key**
V-3 **Simplifying Contaminants Analysis – The Very Latest Developments in GC and LC MS Technology for Routine Screening and Quantitation of Contaminants in Complex Samples**
Walter Hammack, Florida Department of Agriculture and Consumer Services
Salvador Lopez, Institute of Food Safety and Health

1:15 – 2:55 pm **Oral Session 2:** **Ballrooms**
Veterinary Drug Residues
Moderator: Perry Martos (University of Guelph, Canada)

1:15 – 1:35 pm **Andre Schreiber** – AB SCIEX, Ontario, Canada
O-5 **Multi-Compound and Multi-Class Veterinary Drug Screening using Accurate Mass LC-MS/MS**

1:40 – 2:00 pm **Michael Filigenzi** – California Animal Health and Food Safety Laboratory, CA, USA
O-6 **Challenges in Veterinary Analytical Toxicology: Phorbol Esters and Bromethalin**

2:05 – 2:25 pm **Maciej Bromirski** – Thermo Fisher Scientific, Breman, Germany
O-7 **Quick and Sensitive Analysis of Multiclass Veterinary Drug Residues in Meat Products and Urine using Fast Chromatography and a Benchtop Orbitrap Mass Spectrometry System**

2:30 – 2:50 pm **Simon Hird** – Food and Environment Research Agency, UK
O-8 **The Analysis of Horsemeat for the Banned Drug Phenylbutazone**

3:00 – 3:50 pm **BREAK, Exhibition & Posters** **Pavilio**
3:05 – 3:50 pm **Poster Session B1** **Pavilion**
Authors of even poster numbers present

4:00 – 5:15 pm **Oral Session 3:** **Ballrooms**
New Approaches to Ensure Quality
Moderator: Sherri Turnipseed (US FDA Animal Drugs Research Center and Denver Laboratory, CO, USA)

4:00 – 4:20 pm **Jerry Zweigenbaum** - Agilent Technologies, Inc., DE, USA
O-9 **Quality Control/Quality Assurance for Qualitative Analysis using High Resolution and Accurate Measurement Mass Spectrometry**

4:25 – 4:45 pm **Stanley Shaffer** – ABC Laboratories, Inc., MO, USA
O-10 **The Use of Radiolabeled Material to Develop, Troubleshoot, and Validate an Analytical Method**

4:50 – 5:10 pm **André de Kok** – NVWA - Netherlands Food and Consumer Product Safety Authority, Netherlands
O-11 **International (EU and CODEX) Method Validation Guidelines and Use of QC-data for on-going Validation**

6:00 pm **Dinner Cruise** **Depart from Hotel Lobby**
Cruise will leave from Dock at 6:30 pm (Isla del sol)

Tuesday, July 22, 2014

8:00 am – 5:00 pm Registration **Grand Palm Colonnade**
7:45 am – 5:00 pm Exhibition & Posters **Pavilion**
7:45 – 8:15 am Early Morning Coffee **Pavilion**

7:15 – 8:15 am **AB SCIEX Breakfast Seminar** **Tarpon Key**
V-4 **What's In Your Food? See the Tools To Accurately Find & Identify Residues and Contaminants - All the Time, Every Time**
André Schreiber, AB SCIEX, Concord, ON (Canada)

8:30 – 10:35 am **Oral Session 4:** **Ballrooms**
Pesticides and Bees: Analysis Tools and Toxicological Effects
Moderator: Brian Eitzer (Connecticut Agricultural Experiment Station, CT, USA)

8:30 – 8:50 am **Chris Mullin** – Penn State University, PA, USA
O-12 **Determination of Pesticide Co-Formulants and Adjuvants in Honey Bee Related Matrices by LC-ESI-MS**

8:55 – 9:15 am **Roger Simonds** – USDA-AMS-National Science Laboratories, NC, USA
O-13 **Pesticide Residue Analysis of Apiculture Samples from 2007 to Present – Challenges, Perspectives, and Insights**

9:20 – 9:40 am **Horacio Heinzen** – Facultad de Quimica, Motevideo, Uruguay
O-14 **Analytical Tools for the Evaluation of Beehives as Environmental Bioindicators**

9:45 – 10:05 am **Daniel R. Schmehl** – University of Florida, FL, USA
O-15 **Lethal and Sub-Lethal Effects of Field-Level Concentrations of Pesticides in *In-Vitro*-Reared Honey Bees (*Apis mellifera L.*)**

10:10 – 10:30 am **Paul Reibach** – Smithers Viscient, MA, USA
O-16 **Understanding Bee Pesticide Relationships: Laboratory to Field to Laboratory Research Efforts**

10:45 – noon **BREAK, Exhibition & Posters** **Pavilion**
11:00 – 11:45 am **Poster Session A2** **Pavilion**
Authors of odd poster numbers present

12:10 – 1:10 pm **Agilent Technologies Lunch Seminar** **Tarpon Key**
V-5 **Tools for Identifying Pesticide Metabolites in Fruits and Vegetables Using Accurate Mass LC/Q-TOF MS**
Jerry Zweigenbaum, Ph. D., Agilent Technologies

My QuEChERS Extraction is going on a Diet!
Joan Stevens, Ph.D., Agilent Technologies

1:15 – 2:55 pm **Oral Session 5:** **Ballrooms**

Updates from State and Government Laboratories

Moderator: Sherry Garris (South Carolina Dept. of Agriculture, SC, USA)

1:15 – 1:35 pm **Marc Engel** – Florida Dept. of Agriculture and Consumer Services, FL, USA

O-17 **Are Our Children at Risk: Lead Concentration in Sweets?**

1:40 – 2:00 pm **Ping Wan** – Office of Indiana State Chemist, IN, USA

O-18 **Coping with a Laboratory Flood: What Happened, What We Learned**

2:05 – 2:25 pm **Yoko Johnson** – Minnesota Department of Agriculture, MN, USA

O-19 **Evaluation of QuEChERS Application on FIFRA Misuse Samples: Report from a Pesticide Workshop Hosted by a State Laboratory**

2:30 – 2:50 pm **Chris Pappas** – USDA, AMS, S&T, Monitoring Programs Division, VA, USA

O-20 **PDP Sampling and Testing to Support Bifenthrin Section 18 for Brown Marmorated Stink Bug Control**

3:00 – 3:55 pm **BREAK, Exhibition & Posters** **Pavilion**

3:05 – 3:50 pm **Poster Session B2**

Authors of even poster numbers present **Pavilion**

4:00 – 5:40 pm **Oral Session 6:** **Ballrooms**

High Throughput Preparation and Analysis Methods

Moderators: Walter Hammack and Mark Crosswhite (FDACS, FL, USA)

4:00 – 4:20 pm **Bruce Morris** – R J Hill Laboratories, Hamilton, New Zealand

O-21 **Use of a Robotic Solid Phase Extraction Clean-up of Quechers Extracts to Give Improved Matrix Removal for Pesticide Residue Analyses by GC-MS/MS and LC-MS/MS**

4:25 – 4:45 pm **Zoe Grosser** – Horizon Technology, Inc., NH, USA

O-22 **An Automated Technique for the Solid Phase Extraction Sample Preparation of Multiple Organochlorine Pesticide Residues from Wine**

4:50 – 5:10 pm **Sheryl Tittlemier** – Grain Research Laboratory, Canadian Grain Commission, Winnipeg, Canada

O-23 **Sampling and Analysis of Grain Cleaning Byproducts for Truly Rapid Screening of the Mycotoxin Deoxynivalenol in Wheat**

5:15 – 5:35 pm **Dan Hengst** – Nutritional Chemistry and Food Safety, Covance Laboratories, WI, USA

O-24 **UHPLC-MS/MS Analysis and Occurrence of Mycotoxins in Phytomedicines**

5:45 – 6:25 pm **Organizing Committee Meeting** Ballrooms

6:30 – 7:30 pm **Bruker Daltonics Evening Seminar** **Tarpon Key**

V-6 **Mass Spectrometric Strategies for Accurate Screening and Quantitation of Chemical Residues**

Joe Anacleto, Bruker Daltonics, Billerica, MA, USA

7:45 pm Beach Volleyball Game On the Beach

Wednesday, July 23, 2014

6:15 am	Beach Walk/Run	On the Beach
8:00 – noon	Registration	Grand Palm Colonnade
745 – noon	Exhibition & Posters	Pavilion
7:45 – 8:15 am	Early Morning Coffee	Pavilion

7:15 – 8:15 am **Thomson Instrument Co. Vendor Seminar** **Tarpon Key**

V-7 **New Sample Preparation Methodology to enable Higher Recovery, and minimize loss of difficult Analytes in Pesticide and Fungicide Panels by LC/MS or GC/MS**

Lisa Wanders, Sam Ellis, and Joe Machamer, Thomson Instrument Company, Oceanside, CA

8:30 – 10:35 am **Oral Session 7:** **Ballrooms**

Perfluorinated Compounds and Emerging Contaminants

Moderator: Lawrence Zintek (EPA, IL, USA)

8:30 – 8:50 am **Frank Dorman** – Penn State University, PA, USA

O-25 **Environmental Forensic Investigation of Drilling Fluids and Hydraulic Fracturing Fluids used in Shale Gas Wells in the Eastern United States**

8:55 – 9:15 am **Leo Yeung** – Department of Chemistry, University of Toronto, ON, Canada

O-26 **Analytical Challenges on Newly Identified Commercial Fluorosurfactants and Extractable Organofluorine in Human Blood**

9:20 – 9:40 am **P. Lee Ferguson** – Department of Civil and Environmental Engineering, Duke University NC, USA

O-27 **Helping Contaminants Emerge: The Role of High-Resolution Mass Spectrometry in Non-Targeted Analysis of Organic Micropollutants**

9:45 – 10:05 am **Larry Zintek** – US EPA Region 5, Chicago Regional Laboratory, IL, USA

O-28 **Quick and Effective Extraction of Perfluorinated Compounds (PFCs) in Water/Sludge/Soil/Biosolid Samples followed by UPLC/MS/MS Analysis**

10:10 – 10:30 am **Paul Yang** – Ontario Ministry of the Environment, Toronto, ON, Canada

O-29 **Quantitative Determination and Targeted Screening of Contaminants of Emerging Concern Using a POCIS Sampler and HPLC-Orbitrap Mass Spectrometry**

10:45 am – noon **BREAK, Exhibition & Posters** **Pavilion**

12:10 – 1:10 pm **Phenomenex Lunch Seminar** **Tarpon Key**

V-8 **A New Perspective in Pesticides Analysis: You Now Have a Choice – GC/LC/Sample Prep**

Kristen Parnell, Phenomenex, Torrance, CA, USA

1:15 – 2:55 pm **Oral Session 8:** **Ballrooms**

General Topics (I)

Moderators: Jon Wong, USFDA-CFSAN, MD, USA and Katerina Mastovska, Covance Laboratories, WI, USA

1:15 – 1:35 pm **Anthony Macherone** – Johns Hopkins University School of Medicine, MD, USA
O-30 **A New Paradigm in Environmental Health Sciences: Using the Exposome to Determine the Cause of Chronic Human Disease**

1:40 – 2:00 pm **Renata Raina-Fulton –** University of Regina, Regina, SK, Canada
O-31 **GC/MS, GC/MS/MS, and LC/MS/MS Methods for the Analysis of Pesticides and Degradation Products in Atmospheric Samples**

2:05 – 2:25 pm **Benjamin L'homme –** University of Liège, Liège, Belgium
O-32 **Miniaturized Sample Preparation and Minimally Invasive Process for Persistent Organic Pollutant Analysis in Humans**

2:30 – 2:50 pm **Christopher Higgins –** Department of Civil and Environmental Engineering, Colorado School of Mines, CO, USA
O-33 **Accumulation of Contaminants of Emerging Concern in Edible Crops**

3:00 – 3:15 pm **Break** **Grand Palm Colonnade**

3:15 – 5:10 pm **Oral Session 9:** **Ballrooms**
General Topics (II)
Moderators: Jon Wong, USFDA-CFSAN, MD, USA and Katerina Mastovska, Covance Laboratories, WI, USA

3:15 – 3:35 pm **Alexander J. Krynitsky – USFDA-CFSAN, MD, USA**
O-34 **Applications of Mass Spectrometry to the Analysis of Chemical Components Found in Botanical Dietary Supplements**

3:40 – 4:00 pm **Kelly Dorweiler** – General Mills/Medallion Laboratories, MN, USA
O-35 **Improved Identification of Pesticides Using Atmospheric Pressure Gas Chromatography Mass Spectrometry**

4:05 – 4:25 pm **Lynda Podhorniak** – USEPA Analytical Chemistry Laboratory, MD, USA
O-36 **A Miniaturized Residue Analytical Method for the Determination of Zoxamide and its Two Acid Metabolites in Ginseng Using LC-MS/MS**

4:30 – 4:50 pm **Elizabeth Tor –** California Animal Health and Food Safety Laboratory System, Toxicology Laboratory, University of California, CA, USA
O-37 **Determination of Freshwater and Marine Toxins in Complex Biological Matrix Types by LC-MS/MS**

4:55 – 5:10 pm **Closing (Poster Awards)** **Ballrooms**

5:20 – 6:30 p.m. **Informal Forum on Mass Spectrometry** **Ballrooms**
Moderators: Walter Hammack and Mark Crosswhite (FDACS, FL, USA)

Thursday, July 24, 2014

User Meetings:

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

POSTERS

Session A (ODD NUMBERED POSTERS P1, P3, P5, etc.)
Authors stand by their posters from 11:05 am – 11:50 am on Monday and 11:00 am - 11:45 am on Tuesday

Session B (EVEN NUMBERED POSTERS P2, P4, P6, etc.)
Authors stand by their posters from 3:05 pm - 3:50 pm on Monday and 3:05 pm - 3:50 pm on Tuesday

P-1 **Pesticide Residues in Fruits - Are Locally Grown and Organic Fruits a Better Choice?**
Liesl Krone, Action Middle School, Granbury, TX, USA and André Schreiber, AB SCIEX, Concord, ON, Canada

P-2 **Spectro-Magnetic and Antimicrobial Studies on Complexes of Ni(II), Cu(II) and Zn(II) with Ethylenedianil of *O*-Hydroxyphenylglyoxal**
Ali Mohammed Yimer, et al.; Department of Chemistry, College of Natural Sciences, Haramaya University, Ethiopia

P-3 **Analysis of Carbendazim in Orange Juice and Wine: Evaluation of Matrix Effects for Dilute-and-Shoot LC-MS/MS**
Helen (Qingyu) Sun, et al.; Bruker Daltonics, Inc., Fremont, CA, USA

P-5 **Screening and Quantitation of About 250 Pesticides in Fruit Juices with Positive/Negative Switching LC/MS/MS**
Zicheng Yang and Louis Maljers, Bruker Daltonics, Inc., Fremont, CA, USA

P-6 **Comparison of Ionization Techniques for the Analysis of Trace-Level Pyrethroid Insecticides by GC/MS/MS**
Ed George, Bruker Daltonics, Inc., Fremont, CA, USA

P-7 **Determination of Illegal Dyes in Spices by QuEChERS and LC-MS/MS Analysis**
Joan Stevens and Derick Lucas, Agilent Technologies, Wilmington, DE, USA

P-8 **Optimizing Recoveries from Challenging Matrices through Unique Modifications to the QuEChERS Method**
Joan Stevens and Derick Lucas, Agilent Technologies, Wilmington, DE, USA

P-9 **Screening for Hundreds of Pesticides by GC/Q-TOF: New Software with a New Exact Mass Library**
Philip L. Wylie, Agilent Technologies, Wilmington, DE, USA and Sofia Aronova, Agilent Technologies, Santa Clara, CA USA

P-10 **Gain Productivity and Increase Data Quality with the GC/MS/MS Pesticide Analyzer**
Jessica Westland, Agilent Technologies Inc., Wilmington, DE, USA

P-11 **Development of an Automated Sample Preparation and Analysis Workflow for the Determination of Mycotoxin Residues in Different Food Matrices**
Oscar G. Cabrices, GERSTEL Inc., Linthicum, MD, USA and André Schreiber, AB SCIEX Concord, Ontario Canada

P-12 **Automated Derivatization, SPE Cleanup and LC/MS/MS Determination of Glyphosate and Other Polar Pesticides in Drinking Water and Agricultural Commodities**
Oscar G. Cabrices, GERSTEL Inc., Linthicum, MD, USA and André Schreiber, AB SCIEX Concord, Ontario Canada

P-13 **Effect of Moisture and Organic Manure on Persistence of Flubendiamide in Soil**
Shaon Kumar Das, ICAR Research Complex for NEH Region, Gangtok, Sikkim, India and Irani Mukherjee, Division of Agricultural Chemicals, IARI, LBS Building, New Delhi, India

P-14 **Determination of Sodium Iodide Symporter (NIS) Inhibitors in Drinking Waters using Ion Chromatography with Conductivity Detector**
Mehmet Fatih Cengiz, Akdeniz University, Food Safety and Agricultural Research Center, Antalya, Turkey and Ayşe Kevser Bilgin, Faculty of Engineering, Department of Food Engineering, Antalya, Turkey

P-15 **Development and Application of an Analytical Method for the Determination of Neonicotinoid Insecticide Residues in Honey Bee-Collected Pollen by LC-MS/MS**
Tom Thompson et al.; Alberta Agriculture and Rural Development, Edmonton, Canada

P-16 **The Determination of 451 Pesticide Residues in Fruits and Vegetables Using Ultra-High Performance Liquid Chromatography and High Resolution Quadrupole Orbitrap Mass Spectrometry**
Willis Chow et al.; Canadian Food Inspection Agency, Calgary, Alberta, Canada

P-17 **Determination of Pesticide Residues in Oilseeds by GC-MS and GC-ECD Utilizing a Modified QuEChERS Approach**
Don Gaba et al.; Grain Research Laboratory, Canadian Grain Commission Winnipeg, Manitoba, Canada

P-18 **Analysis of Mycotoxins in Cereals Using a Simple Extraction and LC-ESI/MS/MS with Fast Polarity Switching and Scheduled MRMs (Multiple Reaction Monitoring)**
Mike Roscoe et al.; Grain Research Laboratory, Canadian Grain Commission, Winnipeg, Canada

P-19 **How to maintain good peak shape of early eluting polar pesticides after the direct injection of QuEChERS acetonitrile extracts during LC-MS/MS analysis**
Simon Hird et al.; Food and Environment Research Agency, Sand Hutton, York, UK

P-20 **ESI/MS/MS Analysis of Neonicotinoid Insecticides in Canadian Prairie Agricultural Wetlands**
Kerry M. Peru et al.; Environment Canada, Water Science and Technology Directorate, Saskatoon, SK

P-21 **Determination of Highly Polar Pesticide Residues in Food of Plant Origin, by an Automated QuPPe Solution**
Tyler Trent et al.; Teledyne Tekmar, Mason, OH, USA

P-22 **Determination of Pesticide Residues in Honey, by an Automated QuEChERS Solution**
Tyler Trent et al.; Teledyne Tekmar, Mason, OH, USA

P-23 **Determination of Atrazine Residues in Egg Samples of *Podocnemis expansa* from the Brazilian Amazon**
Valdemar Luiz Tornisielo, et al.; Center of Nuclear Energy in Agriculture CENA - University of São Paulo, São Paulo, Brazil

P-24 **Determination of Methyl Parathion Residues in Egg Samples of *Podocnemis expansa* from the Brazilian Amazon**
Franz Zirena Vilca, et al.; Center of Nuclear Energy in Agriculture CENA - University of São Paulo, São Paulo, Brazil

P-25 **Remobilization of Bound Residues of Herbicides in Soils Cultivated with Sugar Cane with Vinasse Application, Straw and Filter Cake**
Marcela L. Viti, et al.; Center of Nuclear Energy in Agriculture CENA - University of São Paulo, São Paulo, Brazil

P-26 **Identification of Metabolites of Study Remobilization of Bound Residues of Herbicides in Soils Cultivated with Sugar Cane with Vinasse Application, Straw and Filter Cake**
Marcela L. Viti, et al.; Center of Nuclear Energy in Agriculture CENA - University of São Paulo, São Paulo, Brazil

P-27 **Evaluation of Environmentally Relevant Concentrations of Florfenicol on Fishes using the Comet Assay**
Rafael G Botelho, et al.; Laboratório de Ecotoxicologia, Centro de Energia Nuclear na Agriculture, Piracicaba, SP, Brazil

P-30 **Validation of chromatographic method for Persistent Organic Pollutants (POP) pesticides in sediment from a recharge area of Guarani Aquifer**
Paulo Alexandre Toledo Alves, et al.; Center of Nuclear Energy in Agriculture CENA. University of São Paulo, São Paulo, Brazil

P-31 **Determination of Atrazine and Methyl Parathion residues in substrate of nests of *Podocnemis expansa* from Brazilian Amazon**
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Allen Misa et al.; Phenomenex, Torrance, CA, USA

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Kory Kelly, Phenomenex, Torrance, CA, USA

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Victor A. Vega et al.; FDA, Southeast Regional Laboratory, Atlanta, GA, USA

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Narong Chamkasem et al.; Southeast Regional Laboratory, U.S. Food and Drug Administration, Atlanta, GA, USA

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Susan Genualdi et al.; US FDA Center for Food Safety and Applied Nutrition, College Park, MD, USA

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Katherine Robbins et al.; US FDA, Center for Food Safety and Applied Nutrition, 5100 Paint Branch Parkway, College Park, MD, USA

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Mark J. Benotti et al.; Battelle Memorial Institute, Duxbury, MA, USA

P-94 **Multi-component quantitative analysis of pharmaceuticals in the environment by UHPLC-MS/MS with on-line SPE**
Robert Clifford et al.; Shimadzu Scientific Instruments Inc., Columbia, MD, USA

P-95 **Analysis of Pesticides in Baby Food Using a Triple-Quadrupole GC/MS/MS, Part II**
Laura Chambers and Robert Clifford, Shimadzu Scientific Instruments, Columbia, MD, USA

P-96 **Screen for Over 470 Residual Pesticides the Same Day your Triple Quadrupole GC-MS/MS is Installed!**
Laura Chambers et al.; Shimadzu Scientific Instruments, Columbia, MD, USA

P-97 **Fast GC-MS/MS Analysis Of Multicomponent Pesticide Residues (360) In Food Matrix**
Moreau Stéphane et al.; SHIMADZU Europe, Duisburg, F.R. Germany

P-98 **Quantitative analysis of pesticides in QuEChERs extracts using APGC/MS/MS**
Kenneth Rosnack et al.; Waters Corporation, 34 Milford, MA, USA

P-99 **Advances in Screening Capability for the Detection of Residues and Contaminants in Food Using Accessible Mass Detection**
Joe Romano et al.; Waters Corporation, Milford, MA, USA

P-100 **Analysis of Glyphosate, Glufosinate and AMPA in Bottled, Tap and Surface Water Using Time De-Coupled Chromatography**
Claude R. Mallet, Waters Corporation, Milford, MA, USA

P-101 **Quantitative Analysis of Pesticide Residues in Rice using LC-MS/MS**
Dimple Shah et al.; Waters Corporation, Milford, MA, USA

P-102 **A Rapid analysis of Sudan And Other Prohibited Dyes In Chili Powder Using Ultra Performance Liquid Chromatography and Tandem Mass Spectrometry**
Dimple Shah and Jennifer Burgess, Waters Corporation, Milford, MA, USA

P-103 **Determination of Pesticide Residues in Oleoresins: Optimized Sample Preparation Prior to LC-MS/MS and GC-MS/MS Analysis**
Michael S. Young and Kim Van Tran, Waters Corporation, Milford, MA, USA

P-104 **Determination of Sudan Dyes in Chili Oleoresin: Optimized Sample Preparation Prior to LC-MS/MS Analysis**
Michael S. Young and Kim Van Tran, Waters Corporation, Milford, MA, USA

P-105 **Discovery of Pesticide Protomers Using Routine Ion Mobility Screening**
Gareth Cleland et al.; Waters Corporation, Milford, MA, USA

P-106 **The Combining of an Integrated Microfluidic Device with Collision Cross Section (CCS) Ion Mobility Screening for the Analysis of Pesticide Residues in Food**
Gareth Cleland et al.; Waters Corporation, Milford, MA, USA

P-107 **Are Pork Residues Present in My Gummy Bears? Gelatin Speciation by LC-MS/MS**
André Schreiber et al.; AB SCIEX, Concord, ON, Canada

P-108 **Target and Non-Target Accurate Mass Screening for Pesticides using LC-MS/MS**
André Schreiber et al.; AB SCIEX, Concord, ON, Canada

P-109 **Identification, Quantitation and Confirmation of Pesticides in Food Samples using LC-MS/MS**
Lauryn Bailey et al.; AB SCIEX, Framingham, MA, USA

P-110 **Routine Targeted Quantitation and Identification of Pesticide Residues using Triple Quadrupole LC-MS/MS and Advanced Scheduling of MRM Transitions**
André Schreiber AB SCIEX, Concord, ON, Canada and Lauryn Bailey, AB SCIEX, Framingham, MA, USA

P-111 **A New Fast and Sensitive HPLC-PDA Method for Analysis of Aflatoxins in Food Products that Eliminates the Need for Post-Column Derivitization**
Jason P. Weisenseel, PerkinElmer Environmental Health, Shelton, CT, USA

P-112 **Advanced Carbon Materials for Sample Preparation of Dioxins, and Furans from Complex Matrices**
Doug Fryer et al.; United Science Corporation, Center City, MN, USA

P-113 **MS/MS^{ALL} with SWATH™ Acquisition for Targeted and Untargeted Pesticide Screening**
Feng Qin et al.; AB SCIEX, Concord, ON, Canada

P-114 **The Use of LC-MS/MS for the Analysis of Allergens in Foods**
Christopher Borton et al.; AB SCIEX, Redwood City, CA, USA

P-115 **Eliminating Matrix Effects and Interferences when Performing High Sensitivity and High Selectivity DMS-LC-MS/MS Pesticide Screening**
Farzad Pakdal et al.; AB SCIEX, Redwood City, CA, USA

P-116 **The Quantitation of Mycotoxins in Cereals using a Simple Sample Extraction and LC-MS/MS using Fast Polarity Switching and MRM Scheduling**
Farzad Pakdal et al.; AB SCIEX, Redwood City, CA, USA

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ORAL PRESENTATION ABSTRACTS

O-1 Science without Borders

Jo Marie Cook

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In 50 years as a residue workshop, the once small FPRW has emerged as NACRW, a major meeting, representing a wide range of residue chemists from countries spanning the globe. This talk will review international participation in our workshop with an emphasis on their contributions to the advancement of science across borders. I'll try to answer the questions "Where have we been? and "Where are we going?"

O-2 Can One Regulation or Analytical Method Fit All Internationally?

Jack F. Kay

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There is constant pressure on food producers to ensure that the food for which they are responsible is safe and wholesome and that consumers are protected from the presence of potentially harmful contaminants. This in turn means that with the increasing importance and reliance on international trade for continued food supplies, producers are obliged to confirm that their goods comply with a wide range of diverse national and international regulations on food safety. Testing may also be required in laboratories using many different analytical procedures, each with their own inherent detection capabilities, specificity, accuracy, etc. Satisfying a set of different requirements could lead to unnecessary duplication of effort on the part of the producers. In addition to this, national and importing authorities may conduct further analytical tests and this may make direct comparison of analytical results obtained from the various sources difficult or invalid. Using veterinary drug residues in food of animal origin as a starting point, this presentation will highlight the steps being taken in this area to harmonise standards and thus protect consumers whilst facilitating national and international trade.

O-3 Multi-residue methods –Complex Performance Criteria Considerations

Anton Kaufmann

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Validation concepts have originally been designed for single residue methods. Such protocols ensured that a particular analyte can be reliably and accurately determined in a particular matrix. Nowadays, multi-residue methods covering a large number of physically and chemically different analytes present in a variety of matrices are being developed. Applying conventional validation protocols for extensive multi-residue methods has become increasingly difficult and time consuming. Furthermore, national or regional validation norms are being used to validate methods that will be employed to monitor globally traded products.

This presentation discusses advantages and disadvantages of current validation concepts. A particular focus is the use of high resolution mass spectrometry based technology. Suggested are ways of how to ensure reliable results for the largest number of possible residues in a variety of matrices. On the other hand, analysts and regulators have to accept the fact that the observed performance criteria for some "naughty" analytes covered by multiresidue methods, will never reach the reliability which is achievable by singe-residue methods. However, most consumers would agree that food safety is more properly enforced by monitoring a multitude of potential residues than by the utilization of one or two extensively validated single residue methods.

O-4 How USDA FSIS Assesses Quality Assurance in Chemistry Methods

Louis H. Bluhm

USDA Food Safety and Inspection Service, Laboratory Quality Assurance Staff, 950 College Station Road; Athens, GA 30605, USA; louis.bluhm@fsis.usda.gov

The USDA Food Safety and Inspection Service (FSIS) is responsible for the safety of US meat, poultry, and egg products. Testing of chemical residues such as veterinary drugs, pesticides, and environmental contaminants such as heavy metals is an important aspect of assuring food safety. FSIS chemistry methods must possess appropriately sound quality assurance and quality control criteria to

support regulatory enforcement of results from the analysis of meat and poultry samples collected by field inspectors at processing establishments across the US. This presentation will cover technical and policy challenges affecting quality assurance in FSIS chemistry methods and the steps that the FSIS laboratory system has taken to address such challenges.

O-5 Multi-Compound and Multi-Class Veterinary Drug Screening using Accurate Mass LC-MS/MS

André Schreiber[1], Nick Zhu[2], Cheng Yuan Cai[2], David Cox[1], Jianru Stahl-Zeng[3]

[1]**AB SCIEX**, 71 Four Valley Drive, Concord, ON, Canada; andre.schreiber@absciex.com; [2]AB SCIEX Shanghai, China; [3] AB SCIEX Darmstadt, Germany

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 AB SCIEX TripleTOF® system 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. In addition, sample-control-comparison was successfully used to find unexpected contaminants. Unknown 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 vision of MasterView™ software.

O-6 Challenges in Veterinary Analytical Toxicology: Phorbol Esters and Bromethalin

Michael S. Filigenzi, Robert H. Poppenga, Linda S. Aston;

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As novel chemicals are produced and familiar ones find new uses, they may end up inside of animals in new and unexpected ways. The field of veterinary toxicology therefore constantly requires new analytical methods. This presentation will describe some challenges encountered in the development of two methods for veterinary toxicological analysis: one for phorbol esters in pet food and the other for bromethalin in animal tissue samples.

Phorbol esters are produced by *Jatropha curcus*. Oil from this plant has been used in the production of biodiesel and there has been concern that the glycerin byproduct of this biodiesel may have made its way into the production of chicken jerky treats. As part of the FDA's ongoing investigation into health effects associated with these jerky treats, we were asked to try to develop an appropriate analytical method. This situation was complicated by the fact that no commercial standards are available for phorbol esters. The approach that was used for the development of this method utilizing high resolution accurate mass spectrometry (HRAMS) and extracts of seeds from *J. curcus* will be described.

Bromethalin is a neurotoxic rodenticide which is commonly available to homeowners. Recently, the EPA has made the decision to significantly reduce the availability of other commonly used rodenticides and it is therefore expected that bromethalin use, along with the attendant inadvertent poisonings of pets and other non-target animals, will increase. We have accordingly developed a method for the detection of desmethylbromethalin, the active metabolite of bromethalin, in animal tissue to aid in the diagnosis of bromethalin toxicosis. The challenging aspect to this method is the fact that analysis of bromethalin standards material by ESI-LC-MS/MS provides somewhat misleading information. The issues encountered with the analysis of these standards and the development of this method will be presented.

O-7 Quick and Sensitive Analysis of Multiclass Veterinary Drug Residues In Meat Products and Urine Using Fast Chromatography and a Benchtop Orbitrap Mass Spectrometry System

Maciej Bromirski[1], Olaf Scheibner[1], Markus Kellmann[1], Charles Yang[2]

[1]**Thermo Fisher Scientific, Bremen, Germany**
[2]Thermo Fisher Scientific, San Jose, CA

A new method, utilizing ultra fast chromatography and a benchtop Orbitrap mass spectrometer is described in this work. The advantage to this approach is a short overall analysis time and a robust method to meet future regulation requirements, offering all options for additional targeted and non-targeted screening.

10 uL injections of extracted meat and urine containing many veterinary drugs were injected onto C18 reverse phase column. All compounds of interest were eluted using one standardized fast gradient elution profile. A bechtop quadrupole Orbitrap mass spectrometer with HESI source was used in positive ionization mode. All method development, data acquisition, data processing and reporting was done using one customized software application.

50 multi-class veterinary drug residues from meat products and waste were analysed in one standardised chromatographic and mass spectrometric method. For quantification, standard curves with eight calibration points were prepared covering the range 10 pg/mL (ppt) to1 µg/mL (ppm). Quasimolecular ions were monitored for quantitation, while additionally up to five fragment ions were monitored for qualification, achieving linear calibration curves over the ranges described above. In this assay, a quick and robust sample preparation method is combined with one short generic analysis method for all compound classes. Together with a very short method development time this new approach stands for high productivity and robustness at the same time. In addition, we could show the potential of this method for additional successful targeted and non targeted screening approaches inside the same data processing software using the same data set.

O-8 The Analysis of Horsemeat for the Banned Drug Phenylbutazone

Simon Hird,[1] Tom Griffith,[1] and Richard Ginn,[1]

[1]**Food and Environment Research Agency**, Sand Hutton York; North Yorkshire, YO41 1LZ, UK; simon.hird@fera.gsi.gov.uk

Phenylbutazone is permitted for use on sport horses and horses kept as companion animals, neither intended for the food chain. There is no maximum residue limit (MRL) or any other action limits set for horses that have received phenylbutazone, so horsed treated with phenylbutazone are not permitted to enter the food chain. Low level residues have been detected during routine monitoring of horse meat from abattoirs for some years. The increased exposure of the UK public to horsemeat via the unwitting consumption of illegally adulterated beef products led to an increase in the frequency of testing quickly followed by a product release scheme for which all results needed to be reported within 48 hours. As phenylbutazone has no MRL set for horse, the very presence of a residue is enough to assign a sample as non-compliant. Modern instruments are extremely sensitive and so detection limits tend to be sub parts per billion concentrations. An analytical method based upon LC-MS/MS was quickly developed, validated and accredited to ISO17025. The results from the surveillance of horsemeat in the UK will be presented.

O-9 Quality Control/Quality Assurance for Qualitative Analysis using High Resolution and Accurate Measurement Mass Spectrometry

Jerry Zweigenbaum

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Good laboratory practices for quantitative analysis is well established, but the development of QC/QA procedures for qualitative analysis has received little attention. This presentation will focus on the need to set both criteria and procedures that will provide assurance that experimental results are both valid and of the highest quality. Using LC/MS with accurate mass measurement as the example the use of internal standards, surrogate standards, and reference compounds will outline the necessary components for both QC and QA. Criteria will be set forth that will encompass the determination of unknown contaminants, non-targeted analysis, and profiling for verification and authenticity. The general concepts of QC/QA for qualitative analysis should provide a basis for development of procedures for most analytical techniques, not just LC/MS.

O-10 The Use of Radiolabeled Material to Develop, Troubleshoot, and Validate an Analytical Method

Stanley R. Shaffer[1], Chris Talken[1], Wesley Fain[1], and Michael Schofield[2]

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[2]Schofield Consulting, Columbia, MO

Radiolabeled 2,4-Dichloro phenoxy butyric ethylhexyl ester (2,4-DB EHE) was used to develop, troubleshoot, and validate a method for analysis in wheat matrices. The material was labeled uniformly on the ring with carbon fourteen ([^{14}C]-2,4-DB EHE). The proposed procedure was to extract the test material with an organic based solvent, hydrolyze the ester to the 2,4-DB acid, remove the matrix with solid phase extraction, and analyze by LC-MS/MS. This presentation will give an overview of how the use of radiolabeled material validated the extraction technique, monitored the effectiveness of the cleanup, and assisted in troubleshooting the loss of 50% of the analyte during the analysis. The reaction of the 2,4-DB acid with a compound in the grain matrix to produce an unknown ester during the procedure would have been next to impossible to detect without the aid of a radioactive tracer. However, with the radioactive tracer the time to troubleshoot the method losses was reduced to only three days.

O-11 International (EU and CODEX) Method Validation Guidelines and Use of QC-data for on-going Validation

André de Kok, Barbara Kiedrowska, Jos Scholten, Marijke de Kroon

NVWA - Netherlands Food and Consumer Product Safety Authority, Laboratory of Food and Feed Safety, National

Reference Laboratory (NRL) for Pesticide Residues in Food and Feed, Wageningen, The Netherlands; a.dekok@nvwa.nl

Method validation is an initial requirement according to ISO-17025 before a laboratory, working under accreditation, can implement an analytical method in routine. The standard validation parameters to be tested are well-known: linearity of detector response, instrument and method limits of detection (LOD, LOQ), matrix effect, accuracy (trueness and precision), specificity/selectivity, robustness and measurement uncertainty. However, the interpretation of how these parameters should be assessed exactly in practice and which method performance criteria should be applied, may differ between continents/countries and application areas. Multiresidue analytical methods, e.g. for pesticides, with applications for a wide range of different matrices, need their specific guidelines in order to be still practical.

In this lecture, the method validation procedures according to the EU Document N° SANCO/12571/2013 will be discussed and compared with some other international guidelines (e.g. CODEX). Validation parameters and performance criteria, procedures for extension of scope of a method (as to new analytes and matrices), under flexible and fixed-scope accreditation, on-going method validation and estimation of measurement uncertainty will also be illustrated with examples from real practice.

O-12 Determination of Pesticide Co-Formulants and Adjuvants in Honey Bee Related Matrices by LC-ESI-MS

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Honey bees are unusually sensitive to organosilicone surfactants, nonylphenol polyethoxylates and the solvent N-methyl-2-pyrrolidone (NMP), widespread co-formulants in agrochemicals and spray tank adjuvants used around beehives. LC-ESI-MS methods for analysis of organosiloxane, nonylphenol (NP) and octylphenol (OP) polyethoxylate surfactants in beehive matrices were developed. A combined liquid-liquid and dispersed solid phase extraction method employing the QuEChERS approach was used on less than 2 grams of honey, pollen or beeswax. For the three trisiloxane surfactants (single polyethoxylate (EO) chain and end-capped with methyl, acetyl or hydroxyl groups), recoveries for each oligomer (2-13 EO_n) were between 66-112% and method detection limits were below 1 ng/g in all matrices. Five honey, 10 pollen and 10 beeswax samples were collected and analyzed. Trisiloxane surfactants were detected in every beeswax and 60% of the pollen samples. Total trisiloxane surfactant concentrations were up to 390 and 39 ng/g in wax and pollen. For analysis of $NP(EO)_{3\text{-}13}$ and $OP(EO)_{3\text{-}13}$ oligomers in bee hive matrices, recoveries were between 75-111%, and method detection limits below 1 ng/g. $NP(EO)_n$ was detected in every hive sample with concentrations up to 10,239 ng/g. Much higher $NP(EO)_n$ residues levels were found in wax followed by pollen than in honey. $OP(EO)_n$ concentrations on average were more than 10 times lower. We are also monitoring NMP and its major degradates in beehive samples. Multi-billion pounds of synthetic organic chemicals used and released into USA environments are formulation ingredients like these, generally recognized as safe, having no mandated tolerances, and whose residues remain largely unmonitored.

O-13 Pesticide Residue Analysis of Apiculture Samples from 2007 to Present – Challenges, Perspectives, and Insights

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The USDA-AMS-National Science Laboratories Gastonia, NC lab (NSL-Gastonia) began analyzing apiculture samples including beeswax, pollen, honey, nectar, bees, and brood as well as related samples like whole flowers and flower nectaries in 2007 in support of university and government researchers delving into the causes of a malady that was given the name “Colony Collapse Disorder” or CCD. NSL-Gastonia adapted the QuEChERS approach to accommodate these unusual and difficult matrices. QuEChERS proved not only to be capable of handling these matrices, but also proved to be very versatile in being easily proportionally micro sized to accommodate small sample sizes, as many apiculture samples received by the lab were less than 1 gram. Since 2007 NSL-Gastonia has analyzed over 9000 samples in support of academic and government researchers as well as private companies and individuals. Today, although pesticide residue exposure has not been singularly correlated as the cause of CCD, the sub-lethal effects of pesticide residues are still being monitored and studied in honey bee colonies to determine what effect they may have on honey bee health as a whole or as indirect contributors to CCD or colony decline. Our experiences, perspectives and insights into the pesticide residue analysis of these samples will be discussed.

O-14 Analytical Tools for the Evaluation of Beehives as Environmental Bioindicators

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Honeybees interact with crops, weeds, woods, water sources and air during their flights; collecting in their bodies along with the desired nectar and pollen, the different contaminants present in the environment. The residues they gather are a primary data source but this information is not enough to indicate that the technological package used in an agricultural zone is responsible for bees´ behavior. It is also necessary to evaluate other compartments of the beehive, like wax, which is an adequate pollution reservoir. Productive and purposefully prepared beehives to be tested as possible bioindicators of the environmental quality of a specific agro-ecosystem were placed in different, studied agricultural and natural areas throughout the country and were sampled. QuEChERS based methods followed by LC-MS/MS analysis of most employed agrochemicals in Uruguay for the analysis of bees and wax were developed and validated. Recoveries ranged from 71 to 119 % with RSDs < 14 % at the LD50 levels of the studied pesticides. LOQs were 0.01- 0.05 mg/kg in bees and 0.01- 0.1 in wax for most of the studied pesticides. Linearity and matrix effects were also evaluated. Beehives contained several residues, e.g.: azoxystrobin, pyraclostrobin, thiacloprid, imidacloprid, tebuconazole, methomyl, carbaryl, haloxifop methyl, coumaphos and hexythiazox at levels ranging from 0.01 to 1.4 mg/kg. Of particular concern are the simultaneous findings of mixtures of fungicides and insecticides, which are 10 fold more toxic to bees than the insecticide alone. The results show that beehives could be used as bioindicators.

O-15 Lethal and sub-lethal effects of field-level concentrations of pesticides in *in vitro*-reared honey bees (*Apis mellifera* L.)

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The decline of pollinators worldwide has been attributed to a combination of factors. Of these factors, pesticides are of particular concern due to the frequency and abundance of residues identified in managed honey bee hives. Over 120 different pesticides have been identified in nectar, pollen, and wax. Here we examine the effects of field-level concentrations of pesticides found in pollen and wax on honey bee survival, development and weight upon adult emergence. Using an *in vitro* rearing technique, we examined the effects of three acaricides (amitraz, coumaphos and fluvalinate), two insecticides (chlorpyrifos and imidacloprid), one fungicide (chlorothalonil) and one herbicide (glyphosate). Each pesticide was integrated into the diet and fed throughout larval development. We found a significant reduction in honey bee survival in bees fed pesticide concentrations found in wax for all seven pesticides and concentrations found in pollen for coumaphos, fluvalinate, amitraz, chlorothalonil, and chlorpyrifos. Bees fed pesticides also displayed several sub-lethal effects. When bees survived until adulthood, we observed delayed pupation and adult emergence when fed amitraz (pollen concentration), chlorothalonil (wax/pollen), chlorpyrifos (wax/pollen), coumaphos (pollen), and fluvalinate (pollen). The weight of adults at the time of emergence was significantly reduced when fed amitraz (wax/pollen), chlorothalonil (wax/pollen), and chlorpyrifos (wax/pollen). Chronic exposure to pesticides during larval development clearly impacts developing bees lethally and sub-lethally. Currently, we are continuing our investigation on the impact of these pesticides on developing bees by measuring detoxification and stress response genes, midgut cell death, and hypopharyngeal gland size at three time points in development.

O-16 Understanding Bee Pesticide Relationships: Laboratory to Field to Laboratory Research Efforts

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Regulators and risk managers are tasked with weighing a compound's ability to control target organisms resulting in a benefit to agricultural productivity versus the potentially detrimental effects on beneficial species. EPA published their scientific opinion in support of the proposed risk assessment of plant protection products on bees. Laboratory studies (Tier 1), such as mortality and larval assays, have use as primary indicators of potential problems. These tests help establish inherent toxicity levels. More complex studies in real environmental settings (Tier 2 and 3) are increasingly being used to quantify exposure taking into account application timing, weather, local environment, individual bee, and hive activity.

Residue analyses in bee-relevant matrices such as-hive media (wax, honey, pollen, bees) and food sources such as plant collected pollen and nectar are used to assess exposure levels under field conditions. These analyses require the development of very sensitive analytical methodology, oftentimes used for the analysis of very small sample sizes. Analysis of individual bees is now quite common.

Samples are analyzed by a variety of methodologies at levels ranging from sub ppb to ppm levels. Honey, pollen, nectar, and bee samples are typically analyzed by HPLC/MS/MS methods. A final comparison of exposure levels detected from field studies and toxicity levels determined from Tier 1 laboratory studies and/or Tier 2 semi-field and field studies are then used for the subsequent risk assessment. Examples of laboratory toxicity testing, field exposure trials, sample analysis, and implications for risk assessment will be presented.

O-17 Are Our Children at Risk: Lead Concentration in Sweet's?

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Honey, syrup and candy samples were collected from retail outlets throughout the state of Florida and analyzed for lead concentration by ICP MS. Lead is a potent neurotoxin and the exposure of developing neurological systems to lead is of special concern. Children absorb lead through the gut at a higher efficiency than adults 40% vs. 10%. A potentially overlooked route of exposure to lead for children is sweets. Honey, syrup and candy samples from domestic and international sources were found to have measurable quantities of lead including some above the FDA regulatory limit of 0.100 ppm. Which products pose the greatest risk to children? Are these risks significant?

O-18 Coping with a Laboratory Flood: What Happened, What We Learned

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In a cold night in January 2014, a 4-inch sprinkler pipe burst in a structure on the top floor of the laboratory building. Water ran through all four levels of the building destroying many parts of the laboratories of the Office of Indiana State Chemist. The pesticide residue laboratory was heavily damaged. This presentation details the laboratory recovery and rebuilding efforts. Experience and lessons learned will be shared.

O-19 Evaluation of QuEChERS Application on FIFRA Misuse Samples: Report from a Pesticide Workshop Hosted by a State Laboratory

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Under the Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA), the U.S. Environmental Protection Agency (EPA) grants the authority over regulating pesticide use to each state. Samples for pesticide misuse cases such as drift complaints are analyzed by state laboratories (state FIFRA labs). One of the challenges the state FIFRA labs face is that residues in misuse samples are often found in metabolite forms. QuECHERS has been applied to a wide range of pesticide classes and has become a staple at laboratories that check compliance of pesticide food tolerances. However, some pesticide metabolites are not analyzed for food tolerance compliance and, therefore, there is very little information about QuEChERS performance on these metabolites. At an EPA sponsored workshop hosted by the Minnesota Department of Agriculture (MDA), spike recoveries of select pesticides and metabolites that are frequently requested for FIFRA misuse cases were evaluated in a mixed vegetation matrix. To our knowledge, the analytes have not been tested with QuEChERS in vegetation matrices. A few different QuEChERS extraction and cleanup salts along with MDA labs' in-house solid phase extraction (SPE) procedure were compared. The result reveals that there is no single extraction/cleanup salt combination that works for all misuse cases but use of QuEChERS with the information from the experiment would help improve efficiency. A brief history and administration of the EPA hosted state workshop (EPA Region 5 Workshop) is also presented.

O-20 Pesticide Data Program Sampling and Testing to Support Bifenthrin Section 18 for Brown Marmorated Stink Bug Control

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PDP collaborated on a project to give Mid-Atlantic apple, peach, and pear famers an additional tool to combat the invasive non-native brown marmorated stink bug (BMSB). The purpose of the collaboration was to provide data to show that the use of the pesticide bifenthrin would not result in harm to consumers while still giving farmers a valuable weapon to protect their crops. Collaborators included the EPA's Health Effects Division (HED); USDA's Interregional Research Project-4 (IR-4), Agricultural Research Service (ARS), and Office of Pest Management Policy (OPMP); PDP; members of the BMSB Working Group; and two testing laboratories that participate in PDP. PDP coordinated the collection of apple, peach, and pear samples by training BMSB Working Group members in PDP sample collection procedures, setting schedules, and providing sampling supplies. The apple and peach sample were collected at participating orchards/farms in Maryland, New Jersey, Pennsylvania, Virginia, and West Virginia

by members of the BMSB Working Group while pear samples were collected at regular collection sites in New York by the same personnel that collect PDP samples. The resulting data showed no bifenthrin residue levels above the EPA proposed tolerance for each fruit. As a result, the EPA approved the emergency Section 18 exemption for use on apples and peaches grown in the Mid-Atlantic States, and significant crop loss was prevented.

O-21 Use of a Robotic Solid Phase Extraction Clean-up of Quechers Extracts to Give Improved Matrix Removal for Pesticide Residue Analyses by GC-MS/MS and LC-MS/MS

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QuEChERS extracts of more difficult sample-types, from fruit with high oil content (e.g. avocado, citrus), to wine, herbs, spices and concentrated food ingredients, can contain high concentrations of matrix that may interfere with instrumental analyses, causing matrix suppression, or instrument fouling. Extract dilution with aqueous buffer for LC-MS/MS can result in precipitation of oils in-vial, taking non-polar analytes out of solution. Dispersive solid-phase extraction (dSPE), is often poor at removing sample matrix, requiring matrix-matching of calibration standards, or large dilutions, which can compromise detection limits. Use of column SPE can give improved clean-up, in part due to the ability to carry out chromatographic separation. Development of a novel robotic SPE clean-up, using miniaturized SPE cartridges, allowed column SPE on an instrument autosampler, with the advantages of precisely controlled flow rates and elution volumes, and clean-up occurring between instrument injections. Custom stationary phase mixtures and elution solvents, gave effective clean-up of spice and concentrated food ingredient samples, resulting in reduced matrix effects compared with dSPE. Spike recovery data for 422 pesticides (including isomers and homologues) on LC-MS/MS showed minimal drop-out of non-polar compounds, and clear diluted extracts, indicating greater removal of oils compared with dSPE, without loss of acidic or quaternary amine analytes. Further experiments with a variety of sample types showed effective removal of interfering sugars, sterols, fatty acids, colors (chlorophyll, carotenoids and anthocyanins), and oils for GC-MS/MS or LC-MS/MS analyses, with acceptable pesticide recoveries compared with solvent-only calibration standards.

O-22 An Automated Technique for the Solid Phase Extraction Sample Preparation of Multiple Organochlorine Pesticide Residues from Wine

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Pesticides are used on agricultural commodities such as table grapes and wine grapes to protect against insects, fungi, mold and other agents that may affect crop yield, cosmetic appearance, and flavor properties. Wine, being a major agricultural commodity, makes it especially important to know the types and quantities of these pesticides that are present in the product as these affect the overall purity, quality and safety to consumers. As a result, many analytical methodologies have been created to monitor these compounds in this food matrix, as dictated by the FDA. Given the number of samples that must be tested in any given year, it is necessary to develop fast, automated SPE methodology for the extraction of these compounds from wine, and also post-extraction treatment of extracts prior to GC/MS analysis.

The established Alcohol and Tobacco Tax and Trade Bureau method developed to manually extract and then measure 20 organochlorine pesticides in wine was optimized for an automated solid phase extraction system using Amino Propyl and HLB cartridges as well as DryDisk membrane technology for drying extracts. The process of method optimization was developed into a flow chart to identify the method steps where optimization was performed and report on the results. The sample preparation optimization flow chart established a scientific process to achieve efficiency of the extraction while demonstrating good recoveries of these 20 organochlorine pesticides. This optimized method demonstrates an improvement to existing manual extraction methodology, and also models an extraction scheme that can be used for other organochlorine pesticides as well as organophosphorous pesticides and antifungal compounds.

O-23 Sampling and Analysis of Grain Cleaning Byproducts for Truly Rapid Screening of the Mycotoxin Deoxynivalenol in Wheat

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Many methods for the analysis of mycotoxins in grain are marketed as appropriate for “rapid” screening. However, this often refers to the detection portion of the analytical method and ignores time required for sampling and sample preparation, which can add a significant amount of time to the testing procedure. Sampling and sample preparation is the area where meaningful reductions in the time taken to test for mycotoxins in grain can be made. Readily available wheat dockage (unthreshed wheat spikelets, chaff, dust,

fragments of stem, etc.) which is a byproduct of grain cleaning and is produced during the process of grain grading in North America, was examined as a novel sample matrix for the rapid analysis of mycotoxins in wheat. Concentrations of the mycotoxins were highest in "light dockage" that contained dust, roughage such as glumes, fragments of stem, or rachis. Mycotoxin concentrations in this matrix were observed at concentrations up to 32 mg/kg deoxynivalenol (DON), 0.532 mg/kg zearalenone, and 0.249 mg/kg ochratoxin A. To examine the feasibility of screening light dockage for DON in a non-laboratory setting, a commercially-available ELISA test kit was evaluated for the analysis of this matrix and compared to the performance of GC-MS. The ELISA was then used to analyze a set of paired light dockage/whole grain samples for DON. Concentrations of DON in light dockage were significantly correlated with concentrations in whole grain, indicating that the light dockage could be used as a readily available matrix for the rapid screening of DON in wheat.

O-24 UHPLC-MS/MS Analysis and Occurrence of Mycotoxins in Phytomedicines

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Phytomedicines, also known as herbal and botanical products, are formulations of various plant materials that are consumed to improve or maintain health. Since these products are derived from plants, the potential exists for contamination by mycotoxins, a class of compounds produced by many different fungal species. When consumed by humans or animals, mycotoxins are linked to numerous adverse health effects. For example, the aflatoxins are carcinogenic, citrinin is nephrotoxic, and the fumonisins are hepatoxic. Other mycotoxins are linked to maladies ranging from vasoconstriction to induced abortion. The occurrence of mycotoxins in phytomedicines may be exacerbated by the methods in which the botanical material is processed. For example, some phytomedicines are the result of an alcohol extraction, which may concentrate the level of mycotoxins in the finished products. With the availability of ultra-high performance liquid chromatography coupled to tandem mass spectrometry (UHPLC-MS/MS), the analysis of multiple mycotoxins in botanical products with nonspecific extractions and minimal purification is possible. However, due to the complex nature of these products, the accurate determination of mycotoxins remains a challenging task. In a survey of various phytomedicines using UHPLC-MS/MS (with electrospray ionization in positive and negative modes), significant amounts of various mycotoxins have been detected. Among the 57 analyzed mycotoxins, enniatins, *Alternaria* mycotoxins, and trichothecenes had the highest incidence of contamination, with levels approaching 40 ppm in a milk thistle product.

O-25 Environmental Forensic Investigation of Drilling Fluids and Hydraulic Fracturing Fluids used in Shale Gas Wells in the Eastern United States

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Recently there has been considerable interest in gas well drilling into the Marcellus shale in the Eastern United States. In states such as Pennsylvania, Ohio and others, there has been a dramatic increase in the development of such wells, but this has not been without controversy. While the gas-drilling industry claims that their processes are completely safe and undamaging to the environment, several environmental groups strongly claim otherwise. Adding to the controversy, the process of hydraulic fracturing of these wells may use various chemicals that are not without concern. The industrial companies claim to self-disclose their formulations, but this is an overstatement, as the exact compositions are not revealed. If a hydraulic fracturing caused some environmental contamination, it would not be easy to determine the source of the contamination due to the lack of disclosure and oversight of the operators. This results in potentially complex environmental forensics analytical method development and sample analysis to determine if a contamination event has occurred, and who the principle polluter may be.

This presentation will address the sampling, sample preparation and analysis of these materials in an effort to develop the chemical compositional understanding of post-drill and/or post-frac fluids so that source identification and source apportionment may be successful in the event of a release. Additionally, this chemical composition may allow for on-site, or near-site bioremediation if the composition is better understood. Various sample preparation strategies will be discussed. GC-TOFMS and GCxGC-TOFMS will be used as determinative techniques due to the inherent advantages over other possible approaches. Finally data will be presented that may allow for the development of protocols to determine both composition of these fluids, and possible point source of pollution should an event occur.

O-26 Analytical challenges on Newly Identified Commercial Fluorosurfactants and Extractable Organofluorine in Human Blood

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Perfluoroalkyl and polyfluoroalkyl substances (PFASs) are anthropogenic chemicals that have been used in various industrial and daily applications since the 1970's. A previous study showed that quantifiable PFASs accounted for approximately 48% on average (range: 30-90%) of the extractable organofluorine (EOF) in Chinese blood collected in 2004, indicating significant amount of organofluorine remained unidentified. Polyfluoroalkyl phosphate diesters (PAPs), perfluoroalkyl phosphonates (PFPAs), perfluorinated phosphinates (PFPiAs), and fluorotelomer sulfonates (FTSAs) are commercial fluorosurfactants that have been newly identified and reported in different environmental matrices. These chemicals might account for a portion of the unidentified organofluorine. In the present investigation, a single LC-MS/MS method using a water/methanol gradient containing 0.1% aqueous ammonium hydroxide was developed to analyze a suite of 48 known PFASs in Chinese whole blood (*n*=47) collected in 2004 and German blood plasma (*n*=112) for the period of 1982-2009. PFPAs and monoPAPs that had been previously reported as suffering severe tailing on a C18 reverse phase column were separated using this method. An ion pair method was used to extract PFASs in blood samples; quantifiable PFASs and EOF were analyzed using LC-MS/MS and Total organofluorine - combustion ion chromatography (TOF-CIC), respectively. Among the newly identified commercial fluorosurfactants, diPAPs: 6:2, 6:2/8:2, and 8:2 and FTSAs: 6:2 and 8:2 were detected in 40% of the German samples, and only 8:2 FTSA was detected in three of the Chinese samples. A mass balance analysis of organofluorine showed that quantifiable PFASs accounted for 48-90% of the EOF, where in German samples, in the periods of 1982-1999 and 2006-2009, quantifiable PFASs were 91% on average (range 63-100%) and 60% (26-100%) of the EOF, respectively. These results suggested that unidentified organofluorines at significant concentrations were present in some cities in China and comparatively higher levels of unidentified organofluorines were observed after year 2000 in Germany.

O-27 Helping Contaminants Emerge: The Role of High-Resolution Mass Spectrometry in Non-Targeted Analysis of Organic Micropollutants

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Recent advancements in high-resolution mass spectrometry (HRMS) and its application to the field of environmental chemistry have for the first time made possible identification of emerging contaminants in complex environmental mixtures without *a priori* knowledge of contaminant identity or occurrence. In this study, we present a strategy and analytical workflow based on LTQ-Orbitrap Velos and Orbitrap Fusion HRMS/MS systems in conjunction with new informatics tools (e.g. data processing, spectral library searching, and literature mining) for identification of emerging environmental contaminants in environmental samples *without* the aid of user-defined molecular databases. In this approach, high resolution (R>100,000) accurate mass (mass error < 2ppm) analyses of water & wastewater extracts are subjected to recursive peak assignment, adduct grouping, isotope pattern scoring, and molecular formula assignment. Chromatographic features and molecular formulas are then filtered using empirical indices (e.g., chromatographic peak shape, mass accuracy, isotopic pattern fit) and heuristic rules for the assignment of probable molecular formulas. Filtered chromatographic features are subjected to confirmation criteria for tentative identification. Utilizing a suite of environmentally relevant compounds, we have established a scoring scheme for assigning the certainty of identification and analytical figures of merit. Importantly, we demonstrate that the developed methodology exhibits low false-positive rates and acceptable false-negative rates for the intended applications. We will discuss our application of these methods to identify emerging contaminants in several important environmental systems, including examination of polar organic micropollutant fate during wastewater treatment and non-potable water reuse.

O-28 Quick and Effective Extraction of Perfluorinated Compounds (PFCs) in water/sludge/soil/biosolid Samples Followed by UPLC/MS/MS Analysis

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The US EPA Chicago Regional Laboratory (CRL) is developing methods for qualitative and quantitative determination of perfluorinated compounds at the low ppt concentration levels. A quick extraction and clean-up procedure using 5 mL water/effluent/sludge samples or a 2 gram soil/bio-solid sample was developed.

One issue with the analysis of common chemicals is minimization or elimination of extraction or preparation steps that may contaminate a sample. For instance, many laboratory products may contain target analytes of interest. Also, PFC compounds are used in automated SPE extraction equipment. The incorporation of state-of-the-art sensitive mass spectrometers allows for the quantification of these analytes at the ppt level with less sample manipulation. These methods for water, sludge, bio-solids and soil are tailored to determine these contaminants in effluents, land applied bio-solids and sludge. Data will be presented in various complex matrices. Many of the methods developed by CRL are incorporated into consensus standards such as the American Society for Testing and Materials (ASTM).

O-29 Quantitative Determination and Targeted Screening of Contaminants of Emerging Concern Using a POCIS Sampler and HPLC-Orbitrap Mass Spectrometry

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Chemicals not previously detected in the environment, or detected at different levels but have not been regulated, are generally referred to as contaminants of emerging concern (CECs). Contaminants of emerging concern represent a diverse group of chemicals that may pose a risk to human health and the environment. Sensitive, selective and reliable sampling and analytical methods are therefore required to monitor CECs in complex environmental matrices. A new analytical workflow for the quantitative analysis of 52 CECs and qualitative screening of more than 378 targeted CECs, including known environmental degradation and treatment by-products, is presented. Field samples collected by a polar organic chemical integrative sampler (POCIS) from surface water and waste water treatment plant effluents were ultrasonically extracted and analyzed without cleanup using a high performance liquid chromatograph (HPLC) coupled to a high resolution Orbitrap mass spectrometer. Extracted ion chromatograms were generated using a mass extraction window of 5 ppm, eliminating matrix interferences observed in HPLC-tandem mass spectrometric analysis. Both quantitative results for the 52 targeted compounds and semi-quantitative data from CECs identified in the qualitative screening can be obtained from one HPLC-MS analysis with good quality control.

O-30 A New Paradigm in Environmental Health Sciences: Using the Exposome to determine the cause of Chronic Human Disease

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The exposome is the summation of all exogenous and endogenous exposure events over the course of individuals' lifetime and is measurable quantity required to understand the causes of most chronic disease. At its essence, the exposome is the compliment to the genome and is comprised of chemical entities found in human blood that are not under direct genetic control. These entities include chemicals derived from the diet, drugs, pollutants, pre-existing diseases and infections and the activity of gut and other microbiota. The tools needed to measure the exposome are those found in integrated biology such as metabolomics and proteomics measurements. One adaptation of the metabolomics workflow is that of exposome-wide association studies (EWAS) of chronic diseases conducted with blood from diseased and healthy subjects. This presentation with define the exposome, EWAS and demonstrate proof of principle for major chronic diseases.

O-31 GC/MS, GC/MS/MS, and LC/MS/MS Analysis for the Analysis of Pesticides and Degradation Products in Atmospheric Samples

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Quantitative methods for the analysis of pesticides and degradation products for atmospheric samples is discussed for a range of pesticide chemical classes including organochlorines, cyclodienes, dinitroanilines, organophosphorus pesticides, methylcarbamates, triazoles, phenylureas, neonicotinoids, and selected pesticides from other chemical classes. Targeted multiresidue methods versus chemical class methods where a larger number of pesticides within a chemical class are generally included will be compared to show difference in selectivity and sensitivities based upon selection of chromatographic and detection system. Options for improved selectivity with choices of columns, mobile phases, MS ionization approaches will be discussed. For some chemical classes where both GC and LC methods can be utilized important sample preparation options including solvent compatibility, and stability of pesticides. For GC methods the use of negative chemical ionization and cold on-column and programmable temperature vaporizer injectors for large volume injections will be highlighted as a means to improve selectivity and sensitivity as well as available options for improving confirmation for dirty samples particularly when the second ion or selectivity monitoring transition for a specific pesticide is weak. We analyzed a wide number of pesticides and degradation products with detection limits typical ranging from 0.1 pg/m^3 to 10 pg/m^3 to assess seasonal variations and atmospheric transport potential from samples collected in the Pacific Northwest and Prairie Agricultural Regions of North America.

O-32 Miniaturized Sample Preparation and Minimally Invasive Process for Persistent Organic Pollutant Analysis in Humans

Benjamin L'Homme, and Jean-François Focant

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Humans all over the world are exposed to chemicals during their life time. Among the thousands of existing anthropogenic compounds are the persistent organic pollutants (POPs). This class of compounds includes polychlorodibenzo-p-dioxins/furans (PCDD/Fs), polychlorobiphenyls (PCBs), organochlorine pesticides (OCPs), and halogenated flame retardants (HFRs). Human biomonitoring of some of those toxic molecules is nowadays typically performed on relatively large samples (5-100mL blood) requiring uncomfortable and badly perceived venipunction for patients. Analysis on small amount (20µL) of blood could, by contrast, be considered as non-invasive since it simply consists in pricking the heel or finger to sample a few drops of blood from patients. However, such small biological samples, either in the liquid form or dried on filter paper (dried-blood spots (DBS)) require the development of specific miniaturized methods for reliable analysis at very low level.

We developed a method for the quantification of persistent organic pollutants (POPs), in the context of the Stockholm Convention, by miniaturized solid phase extraction using MEPS (micro-extraction by packed sorbent) and GC-MS/MS. Samples consist in 20µL of liquid serum as well as 20µL dried-blood spots. The study aims to push miniaturization to its limits; indeed, a maximum of 150µL of solvents are needed for the whole procedure. Results were compared to those obtained with classical analysis procedure including GC-HRMS methodology.

O-33 Accumulation of Contaminants of Emerging Concern in Edible Crops

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Anthropogenic organic contaminants present in wastewater and sewage sludge may persist through wastewater treatment and raise concerns when reclaimed water becomes a viable resource for irrigation or when wastewater-derived biosolids are land applied as a soil amendment. Contaminants of emerging concern (CECs) reach wastewater streams following human use and include but are not limited to pharmaceuticals and personal care products (PPCPs), flame retardants, corrosion inhibitors, plasticizers, and perfluoroalkyl acids (PFAAs). All of these CECs have the potential to bioaccumulate in crops, and robust analytical methods are needed to assess the potential contamination of food crops. Controlled greenhouse experiments designed to examine the potential bioaccumulation of selected CECs into representative edible crops from reclaimed water as well as from biosolids amended soil were performed. Strawberry and lettuce plants were grown with tap water, reclaimed water, and reclaimed water fortified with increasing levels of CECs. Four additional crops (tomato, radish, celery, and peas) were grown in biosolids-amended and control soils. Analysis of plant tissues for the suite of target CECs allowed for the calculation of bioaccumulation factor (BAF) values in each edible plant tissue. Additionally, non-edible portions were analyzed to improve our understanding of how CECs will be transported and accumulated within plants. Results from this study have important implications with respect to the potential exposure of humans to contaminants in fresh produce. The ultimate goal of this ongoing project is to improve mechanistic understanding of plant uptake of CECs, thereby allowing for advancement of models intended to predict human exposure.

O-34 Applications of Mass Spectrometry to the Analysis of Chemical Components Found in Botanical Dietary Supplements

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A variety of herbal dietary supplements are currently available and some consumers believe that their use may benefit in the management of their health and provide a safer alternative to conventional medical treatment. It is well documented that accidental or intentional substitution of intended botanical ingredients with cheaper more readily available plant materials may result in adulteration of the final product with natural ingredients that may be toxic and this may cause serious health problems to consumers. The first part of the presentation covers an ultra-high performance liquid chromatography-quadrupole-orbital ion trap mass spectrometry method for the determination of 96 pharmaceuticals, plant toxins, and other plant secondary metabolites in herbal dietary supplements. Target analytes were extracted from samples using the QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) procedure. The instrument was operated in full MS-data dependent tandem mass spectrometry acquisition mode which enabled collection of quantitative high resolution full MS data and confirmatory high resolution data in a single run. Because of matrix variations and difficulty in obtaining a blank matrix for suitable matrix matched standard experiments, the method of standard additions was among the methods applied for quantitation. The second part of the presentation discusses the adulteration with phenylethylamine-type compounds in weight loss and body building supplements. An independent GC-MS method was used for comparison to the original LC-MS/MS method, to confirm the presence of β-methylphenethylamine and the absence of amphetamine in dietary supplements containing *Acacia rigidula*. Other examples on how to best utilize mass spectrometry will be shown.

O-35 Improved Identification of Pesticides Using Atmospheric Pressure Gas Chromatography Mass Spectrometry

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Successful testing of pesticides in food matrices requires techniques permitting fast and reliable determination of residues, and

the absence thereof. Mass spectrometry offers residue chemists the ability to discriminate against thousands of peaks, identifying trace level residues in a variety of food matrices. Electron impact (EI) ionization in gas chromatography mass spectrometry provides highly specific data for trace level pesticides. Mass fragments unique to the compound provide added confidence when reviewing mass spectral data. However, as the size of the fragment decreases, so do the selectivity and sensitivity, resulting in difficult data interpretation, particularly at low part per billion levels. False negatives may be reported if the fragments are too small to be detected. Furthermore, co-eluting matrix interferences present in sample extracts obscure targeted peaks in the chromatography and may contribute to false positive determinations. All factors contributing to the complexities described increase data analysis time.

Atmospheric pressure ionization gas chromatography (APGC) provides a softer ionization of the analyte, thereby preserving the molecular ion and heavier mass fragments for better identification. This technique coupled with high resolution quadrupole time of flight mass spectrometry, produces both low and high energy spectra. Comparisons between EI and APGC ionization for several classes of pesticides, including pyrethroids will be presented. Various matrices, including flaxseed, dried soybean, and corn gluten feed were evaluated. Lower mass fragments were primarily produced in the high energy spectra, whereas in most cases low energy spectra successfully generated the molecular ions for improved identification.

O-36 A Miniaturized Residue Analytical Method for the Determination of Zoxamide and its Two Acid Metabolites in Ginseng Using LC-MS/MS

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A miniaturized residue method was developed for the analysis of the fungicide, zoxamide, and its metabolites in ginseng. The zoxamide metabolites, 3,5-dichloro-1,4-benzenedicarboxylic acid and 3,5-dichloro-4-hydroxymethylbenzoic acid, which are small acid molecules, could not be extracted from the ginseng matrix with common multi residue methods. The extraction method to be presented effectively and rapidly recovers both the zoxamide parent compound and its acid metabolites from ginseng. In addition, this method avoids the use of derivatization of the small acid molecules by using LC-MS/MS instrumental analysis. In a quantitative validation at three levels for zoxamide, ranging from LOD to 10X LOQ, and five levels, ranging from LOD to 30X LOQ, for the metabolites, acceptable performances were achieved with overall recoveries of 70-120% and <20% RSD for the three analytes.

O-37 Determination of Freshwater and Marine Toxins in Complex Biological Matrix Types by LC-MS/MS

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Toxin-producing harmful algal blooms (HAB) are increasing in frequency in both fresh and marine waters. Several cases of intoxications of various animal species due to HAB toxins were diagnosed at the CAHFS-Toxicology Laboratory. Anatoxin-a intoxications were the most common among dogs, while microcystins, domoic acid and tetrodotoxin among wildlife. Cattle deaths were associated with water sources contaminated with microcystins. Various samples of drinking water, feed, GI contents, urine, serum, bile and tissues from suspected poisoning cases were analyzed by LC-MS. In clinical situations a quick diagnosis is essential, and a slow, typically used freeze-thaw process needed to disrupt the cyanobacterial cell walls needed to be eliminated. Efficiency of the sonication process using a horn, 400 W sonifier for cell wall disruption in a lake water sample containing a microcystin-LA containing matts of *Microcystis aeruginosa sp.* was compared with freeze-thaw cycles. Sonication over five minutes was proven to be sufficient for complete cell wall disruption and released similar amounts of microcystin-LA as five freeze-thaw cycles. Spiking experiments proved the stability of MC-LA, LR, YR, RR and anatoxin-a during the sonication. The LC-MS methodology routinely used in the toxicology laboratory was a successful tool for unambiguous diagnosis of intoxications in a variety of poisoning cases. Diagnosis of animal poisonings due to HAB toxins has dramatically improved since the development of LC-MS methods.

POSTER ABSTRACTS

P-1 Pesticide Residues in Fruits - Are Locally Grown and Organic Fruits a Better Choice?

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There is a lot of concern about how clean the fruits are that are coming from outside the United States. The U.S. EPA regulates what pesticides are used domestically but how much to do we monitor the chemicals being used on imported products? I will evaluate which fruits are freer of pesticides, imported fruits or locally grown ones.

Assorted fruits (local and imported, organic and non-organic) were blended and extracted using QuEChERS and analyzed by LC-MS/MS.

Calibration curves and QC results were obtained for ~ 180 pesticides. Calibration curves all have an R value between .998 and .999 which means they are a good fit. The QC results for the 10ppb standard are also good. The accuracies, which are a measure of how close the calculated value of the standard is to the actual value, are all between 81% and 105%. The Percent Coefficient of Variance (CV) should be less than 10%, and this was observed.

Seven of the fruit samples contained detectible amounts of pesticides. Pesticides were pretty evenly distributed between the local and imported fruits. The US Grapes contained several detectible pesticides whereas the Grapes from Chile and Peru did not. The dirtiest sample appears to be the Organic Pear from the US! It contains several non-organic pesticides including Thiabendazole which is a fungicide. Organic US cherries and cherries from Chile were analyzed. The organic cherries are truly organic since only chemicals that are approved as organic were detected. The most significant result for the Chile cherries was the level of Tebuconazole reported! Tebuconazole was detected is below the maximum allowable limit. US grown organic pear was compared to a Korean pear. The US pear was relatively dirty. It does contain the organic Spinosyn A and D pesticides but also contains several hits for non-organic pesticides - all are below the maximum allowable limit. In this example it is better to eat an imported pear!

P-2 Spectro-Magnetic and Antimicrobial Studies on Complexes Of Ni(II), Cu(II) And Zn(II) With Ethylenedianil Of *O*-Hydroxyphenylglyoxal

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The Schiff's base ligand and its complexes with Ni(II), Cu(II) and Zn(II) were synthesized and characterized by elemental analysis molar conductivity, infrared and ultraviolet-visible spectroscopy and magnetic susceptibility measurements. The ligand, ethylenedianil of o-hydroxyphenylglyoxal, $C_{18}H_{16}N_2O_4$, has been synthesized by condensation of ethylenediamine and orthohydroxyphenylglyoxal. The metal complexes were prepared by mixing saturated solutions of ligand and metal salts in appropriate molar ratio in acetone and methanol solvents. The study also confirmed the formation of mono-, di- and trinuclear isopolystructures of the complexes in square planar geometry except for Zn(II) which has tetrahedral stereochemistry. The synthesized ligand and its metal complexes were screened for their antimicrobial activities against two bacterial strains, Staphylococcus aureous and xanthomonas holcicola and two fungal strains, Aspergillus niger and Fusarium oxysporum using disc diffusion method, showing that the complexes were of more antimicrobial activity than the free Schiff's base

P-3 Analysis of Carbendazim in Orange Juice and Wine: Evaluation of Matrix Effects for Dilute-and-Shoot LC-MS/MS

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Carbendazim, a banned fungicide in the US was detected in orange juices imported from Brazil (2011) and in Chinese brand wines (2012), which has triggered the high demands for carbendazim screening in juice produces. LC-MS/MS is a powerful technology for trace level pesticide analysis in food. Sample preparation of pesticide analysis in juice generally employs two strategies: solid phase extraction (SPE) or dilute-and-shoot. Dilute-and-shoot is preferred due to its simplicity, however no comprehensive study has yet been done to evaluate dilution-factor-caused matrix interference for carbendazim analysis. In the current study, an quantitative

MRM method for carbendazim analysis in orange juice and wine matrices was developed using a Bruker EVOQ™ LC-MS/MS system with a calibration range from 0.005 up to 50 ppb (ng/mL). Good instrument sensitivity and robustness were achieved during method development. Matrix effect was evaluated by comparing matrix effect% against during dilution factors. Our results show that orange juice and wine matrices exhibit different matrix effects and provide the scientific reference to determine the appropriate dilution factor for carbendazim analysis in juice and wine. Finally, the diluted orange juice and wine samples were tested by EVOQ LC-MS for carbendazim screening.

P-5 Screening and Quantitation of About 250 Pesticides in Fruit Juices with Positive/Negative Switching LC/MS/MS

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Liquid chromatography coupled with tandem mass spectrometry operated in multiple reaction monitoring (MRM) mode with electrospray ionization (ESI) is widely used for polar, semi-volatile, and thermally labile pesticides in food testing. Many contract labs currently perform multi-residue analysis of pesticides using separate positive and negative methods due to instrument limitations especially for methods with hundreds of MRM transitions. This requires twice the sample and twice the analysis time. Recently, the Bruker EVOQ Elite LC-triple quadrupole system has been introduced to the market; thereby providing fast positive/negative switching allowing for simultaneous determination of positive and negative co-eluting compounds numbering in the hundreds.

A study using the EVOQ analyzed about 250 pesticides in apple juice, cranberry juice, grape juice, orange juice and V8 vegetable juice using only one method with positive negative switching for about 500 MRM transitions. The measurements were conducted by dilute-and-shoot without sample enrichment. The fruit juices were centrifuged and diluted 10-fold before injection. An YMC-Pack ODS-AQ, 3 µm, 150 mm x 3 mm (I.D.) column with mobile phases (A) 5 mM ammonium fluoride in water, and (B) methanol were used. The total run time was 18 minutes including re-equilibration. The preliminary results showed that both positive and negative co-eluting peaks have R^2 >0.99 with linear range 0.1 to 100 µg/L.

P-6 Comparison of Ionization Techniques for the Analysis of Trace-Level Pyrethroid Insecticides by GC/MS/MS

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Pyrethrins are natural insecticides derived from chrysanthemum flowers, and were used as the basis to create a class of synthetic pyrethroids. These pyrethroids are stable, toxic, and are more effective against a broader range of pests. They make their way into the environment through agricultural use and domestic control of mosquitos, and tend to persist in soils and sediments. Run-off from residential sources contributes to surface water and waste water treatment plants. State agencies such as the California Department of Food and Agriculture and others have implemented monitoring programs requiring low to sub-part-per-billion reporting limits in soil and water samples.

Tandem GC/MS is an ideal technique to analyze for the synthetic pyrethroids, because it can discriminate effectively against matrix and provide trace level detection. There have been several publications that have evaluated both ion trap and triple quadrupole instruments with either electron or chemical ionization. In many cases, relatively large sample pre-concentration and/or large volume programmed temperature vaporization (PTV) injections have been used to reach the required detection limits.

The Bruker SCION triple quadrupole mass spectrometer has a unique axial ion source and lens free design, resulting in robust operation and excellent sensitivity for the pyrethroid insecticides. In this work, electron and chemical ionization techniques are optimized and compared in terms of calibration range, method detection limits, and precision. A standard hot splitless injection is used which reduces instrument matrix load and is far simpler than PTV. Discrimination against matrix components resulting from surface and waste water extracts will also be evaluated.

P-7 Determination of Illegal Dyes in Spices by QuEChERS and LC-MS/MS Analysis

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A wide range of synthetic dyes are used in industry for coloring various materials such as textiles, waxes and leather garments. The use of these dyes in food is not allowed because of health concerns (cancer-risk) related to their intake. The market takes a view that something like paprika or chili has an ideal color and that it is only possible to get the best price if it's the perfect match. Foreign producers are deciding to use inexpensive illegal dyes to achieve the perfect color, which in turn boosts the price for the spices and their profit. Spices under suspicion are huge part of American and British diets because of the surge in popularity of Indian, Chinese, Thai and Mediterranean dishes.

The QuEChERS methodology and a superficially porous LC column were employed to extract a series of illegal dyes. The QuEChERS procedure offers a simple sample preparation technique for the extraction of illegal dyes from difficult sample matrices like spices. The use of a superficially porous LC column is extremely well suited for use with more complex samples, with efficient mass transfer. The combination of QuEChERS and superficially porous column equates to faster analysis time, higher throughput with optimum results.

P-8 Optimizing Recoveries from Challenging Matrices through Unique Modifications to the QuEChERS Method

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QuEChERS is a well-known, established method, often implemented as a go to sample preparation technique for extraction of multiple classes of compounds from fruits and vegetables with analysis by GC- or LC-MS/MS. Since its inception in 2003 and later validated as AOAC 2007.01 and EN 15662 the method has seen an active expansion into other matrices for instance meat, fish, oils, teas, flour, spices, grains and processed foods to name just a few. Implementing the QuEChERS method in non-fruit and vegetable matrices can be quite challenging since the matrix can contain high percentages of lipids, proteins, additives and concentrated components not found in fruits and vegetables. Since QuEChERS is a non-exhaustive sample preparation technique; as the complexity of the matrix increases so can the amount of residual matrix components remaining in the analysis extract. Although analysis by MS/MS will hide the presence of these matrix components, they are still present in the extract and will have adverse effects on analyte recovery, reproducibility, and method ruggedness; not to mention the detrimental effects to the instrument flow path and increased maintenance.

Modifications to the QuEChERS method have been noted in the literature to address the issues associated with complex matrices. Modifications range from changes in extraction solvents to the addition of various sorbents in the d-SPE. Trying to determine the approach one should implement with a new sample matrix can be quite a daunting task and require trial and error. In this application we will present a systematic approach to determine and choose optimization conditions/additions based on the analytes and sample matrix. This offers a simple and straight forward approach to optimize the QuEChERS methodology for more complex matrices without loss of analyte and additional sample

P-9 Screening for Hundreds of Pesticides by GC/Q-TOF: New Software with a New Exact Mass Library

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Most laboratories use GC/MS/MS methods for the analysis of volatile pesticides in foods because GC triple quadrupole instruments offer high sensitivity and selectivity. But GC/MS/MS is a targeted approach and only those compounds on the target list will be found. A complimentary screening method is needed to look for a much larger number of pesticides. This paper discusses the use of a high resolution accurate mass (HRAM) quadrupole time-of-flight MS (GC/Q-TOF) to screen for about 700 pesticides. In the TOF mode, HRAM spectra are acquired over the instrument's full mass range. Identification of analytes is possible when two or more characteristic accurate mass ions are found at the correct retention time. In theory, one could screen for an unlimited number of compounds so long as their characteristic accurate mass ions and retention times are known.

This presentation introduces an "All Ions" workflow for the screening of pesticide residues in foods using a GC-Q/TOF in the electron impact ionization mode and a new exact mass spectral database containing ~700 pesticides. The all ions software automates screening by choosing characteristic exact mass ions for each compound and extracting them from the chromatogram. It then looks for a molecular ion and, if found, compares its isotope pattern the theoretical one. Then it looks at the co-elution profiles of fragment ions, creating a co-elution plot and co-elution score to help visualize and express the covariance of the extracted accurate mass ions. A table summarizes the results and indicates if the pesticide is present.

P-10 Gain Productivity and Increase Data Quality with the GC/MS/MS Pesticide Analyzer

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Pesticide residue analysis is a complex task requiring the analyst to search for dozens, or even hundreds, of compounds in a wide variety of crop or environmental matrixes. This also requires a lab to have a completeness of MS/MS MRM transitions in an acquisition method. A triple quadrupole GC/MS (GC/MS/MS) provides excellent sensitivity and selectivity in analyzing complex matrixes; and the use of a developed MRM database offers an increase in analysis productivity.

Utilizing an Agilent 7890B GC and 7000C Series Triple Quadrupole GC/MS system, with a newly developed pesticide residue method and the Pesticides and Environmental Pollutant (P&EP) MRM not only simplifies a lab's startup process, but it also helps to increase their productivity and throughput. The new method focuses on the ability to acquire multiresidue pesticide analysis of large target lists in complex matrices including, apple, pepper, and kale. The new Graphical User Interface (GUI) allows for a more streamlined approach for developing an MRM acquisition and/or quantitation method. Using this new GUI, the development of an MRM acquisition method and a quant method can be effortlessly created in less than 5 minutes.

P-11 Development of an Automated Sample Preparation and Analysis Workflow for the Determination of Mycotoxin Residues in Different Food Matrices

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The abundance of mycotoxins in food products is a major concern in product safety due to the health risks they pose to humans and livestock. Mycotoxin levels in food and animal feed are regulated in most countries and there is great interest in a fast, sensitive, and selective analysis method. Determining mycotoxin concentrations at trace levels in the presence of large amounts of sample matrix is a challenging task, making the accuracy and precision of the analytical results fundamentally dependent on the sample preparation methodologies used to isolate the mycotoxins from the complex food and animal feed matrices. In this report, we describe an automated sample preparation and analysis workflow for the screening of multi-mycotoxin residues in different food matrices (corn, wheat, rice) by LC/MS/MS. The extraction methodology was performed using a GERSTEL MPS robotic autosampler interfaced to an AB SCIEX QTRAP® 4500 LC/MS/MS System. The automated sample cleanup and analysis workflow targeted a panel of 14 mycotoxins (aflatoxins, trichotecenes and fuminosins). The LC/MS/MS was operated in Multiple Reaction Mode (MRM) with fast polarity switching for accurate detection. Dependent MS/MS spectra were also acquired in the Enhanced Product Ion (EPI) mode after being triggered from a Scheduled MRM™ Information Dependent Acquisition (IDA) survey scan. The automated SPE cleanup procedure provided extraction efficiencies greater than 70% for all mycotoxins screened in the different food samples with RSDs less than 15%. In addition; good linearity was achieved (R^2 values of 0.98 or greater) reaching limits of quantitation lower than the action levels established by the FDA for most analytes. The ability to automate Solid Phase Extraction clean-up methodologies focused on mycotoxin sample extracts and to couple the extraction directly to LC/MS/MS allows compound identification with highest confidence based on mass spectral library matching, and improves laboratory productivity by streamlining the complete analytical process.

P-12 Automated derivatization, SPE cleanup and LC/MS/MS determination of glyphosate and other polar pesticides in drinking water and agricultural commodities

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Glyphosate (N-phosphonomethyl glycine) and glufosinate [ammonium (S) -2-amino-4-[hydroxyl (methyl) phosphinoyl] butyrate] are non-selective post emergence herbicides used for the control of a broad spectrum of grasses and broad-leaf weed species in agricultural and industrial fields. Aminomethyl-phosphonic acid (AMPA) is the major metabolite of glyphosate and also included into the pesticide residue definition. According to recent reports, there has been a dramatic increase in the usage of these herbicides which are of risk to both human health and the environment. Therefore, there is a global need for reliable and sensitive methodology to determine trace level residues from these pesticides in water and food.

In this presentation, we describe an automated workflow to derivatize and analyze water and food samples for glyphosate, glufosinate and AMPA by LC/MS/MS using a GERSTEL Multi Purpose Sampler (MPS) 2XL configured with an online solid phase extraction module (SPE^{XOS}) coupled to an AB SCIEX QTRAP® 4500 system. The SPE^{XOS} system uses small cartridges packed with 10-50 mg of sorbent, which allows elution to be carried out with small volumes of solvent resulting in high throughput sample cleanup. The total cycle time per sample for the automated sample derivatization and online SPE was approximately 25 minutes, enabling "just in time" sample preparation using the GERSTEL MAESTRO software.

Water samples were injected directly into the LC/MS/MS system providing sufficient sensitivity to identify and quantify targets at sub 100 µg/L concentrations. Food samples (corn and soy) were initially extracted either by a modified batch equilibrium technique using or the QuPPe method followed by automated derivatization and cleanup using online SPE-LC/MS/MS analysis. The results using the QuPPe extraction where compared to results obtained when using the batch equilibrium procedure obtaining recoveries between 70-120% for both matrices. All target compounds were easily identified and quantified at 10 µg/kg levels with %CVs less than 10% in most cases.

P-13 Effect of Moisture and Organic Manure on Persistence of Flubendiamide in Soil

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Persistence of flubendiamide in soil as affected by moisture and organic manure was studied. The present study reports persistence of flubendiamide [N^2-{1,1-dimethyl-2-{methylsulfonyl)ethyl}-3-iodo-N^1-{2-methyl-4-{1,2,2,2-tetrafluoro-1 (trifluoromethyl)ethyl} phenyl}-1,2-benzene dicarboxamide] in a sandy loam soil. Dissipation for the pesticide followed mono-phasic first order kinetics. The persistence of flubendiamide was more in dry soil followed by field capacity and submerged condition with half life values of 150.5-158.4 days for submerged soil, 177.0-181.1 days for field capacity soil and 206.6-215.0 days for dry soil. It was found that there is slight effect of fortification level on dissipation of flubendiamide in soil. In all the cases i.e. dry, field capacity and submerged condition dissipation was slightly slower at 10 µg g^{-1} level. Amendment of organic manure (2.5%) to the soil enhanced the degradation of the insecticide, and the half-life values in field capacity and submerged soils were 155.1 and 130.8 days, respectively.

P-14 Determination of Sodium Iodide Symporter (NIS) Inhibitors in Drinking Waters using Ion Chromatography with Conductivity Detector

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Excessive usage of agricultural inputs such as fertilizers and pesticides is well known to possible endocrine related health problems. Recent health statistical data shows that health problem due to thyroid gland disruption, also known as goiter, has gained a great acceleration within the last decade. It was reported that the residues of NIS inhibitors in water and food samples can result in goitrogenic effects. The most effective inhibitor compounds were stated as perchlorate, thiocyanate and nitrate. In this research; a sensitive, rapid and reliable method using Ion Chromatography with conductivity detector to determine NIS inhibitor ions in drinking waters was improved. The method involves measurement of conductivity for the calculation of dilution factor, filtration through 0.45 µm syringe filter and injection to the system. No sample pre-treatment and concentration steps were required prior to the injection. Various analytical parameters associated with mobile phase, column and detector were evaluated according to peak shapes, retention times, capacity factors and resolutions. In addition, the effects of some possible interferences were reviewed. In conclusion, optimum analytical conditions were established. Using this method, the limit of detection and limit of quantification were obtained as 2.41 µg/L and 8.03 µg/L for perchlorate; 8.96 µg/L and 29.89 µg/L for thiocyanate; 257.47 µg/L and 858.23 µg/L for nitrate, respectively. Based on the evaluation results it can be stated that the method developed could be regarded as a new alternative method for routine analysis of trace levels of NIS inhibitors in drinking water samples.

P-15 Development and Application of an Analytical Method for the Determination of Neonicotinoid Insecticide Residues in Honey Bee-Collected Pollen by LC-MS/MS

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Neonicotinoid insecticides have recently come under considerable scrutiny regarding their potential contribution to increased honey bee colony losses. Four neonicotinoids (acetamiprid, clothianidin, imidacloprid, and thiamethoxam) are registered for use in agriculture in Alberta. In order to investigate the environmental contamination of apiaries resulting from the transportation of pollen into the beehive, pollen samples were collected using traps set up at colony entrances. In the analytical method, pollen samples were fortified with a standard solution containing deuterated analogues of each neonicotinoid insecticide prior to extraction with acetonitrile. Cleanup of the pollen extracts consisted of partitioning with hexane followed by dispersive solid phase extraction using a mixture of octadecylsilane (C18) and primary-secondary amine (PSA). Extracts were analyzed by LC-MS/MS using multiple reaction monitoring. Matrix-matched calibration standards ranging from 0.5 to 25 µg/kg were employed along with internal standardization using the deuterated analogues. Detection limits were 0.2 µg/kg or lower for all analytes. A total of 73 pollen samples were collected throughout the province of Alberta. Clothianidin and thiamethoxam were the most frequently detected neonicotinoid insecticides (16 and 17 samples respectively). Acetamiprid was detected in only 2 of 73 samples while imidacloprid was not detected. Residue levels were very low with the majority of the samples containing less than 1 µg/kg of neonicotinoid insecticides.

P-16 The Determination of 451 Pesticide Residues in Fruits and Vegetables Using Ultra-High Performance Liquid Chromatography and High Resolution Quadrupole Orbitrap Mass Spectrometry

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This poster presents the application of an Ultra-high Performance Liquid Chromatography (Thermo Hypersil GOLD, 1.9 µm, 100 mm × 2.1 mm column) and High Resolution Electrospray Ionization, Quadrupole-Orbitrap Mass Spectrometry (UHPLC/ESI Q-Orbtrap MS) method for the screening and quantification of up to 451 pesticide residues in fruits and vegetable matrices. Pesticides were extracted and cleaned-up from matrices with a modified QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) method. Quantification was achieved using matrix-matched standard calibration curves with isotopically labelled pesticides as internal standards. Analytical range is 5 to 500 µg/kg, and quadratic regression and 1/x weighting were used. Method performance was characterized by overall recovery, intermediate precision and measurement uncertainty. In fruits, about 94% of the pesticides had recoveries between 81 and 110%, 99% had an intermediate precision ≤ 20% and 90% showed measurement uncertainty ≤ 20%. In vegetables, about 91% of the pesticides had recoveries between 81 and 110%, 99% had an intermediate precision ≤ 20% and 80% showed measurement uncertainty ≤ 20%. All performance numbers were evaluated according to a statistically designed experiment, i.e. nested design via SAS. By exploiting high chromatographic column efficiency and high mass resolution, this method was able to analyse 451 pesticide compounds in a 14 minute, single injection run.

P-17 Determination of Pesticide Residues in Oilseeds by GC-MS and GC-ECD Utilizing a Modified QuEChERS Approach

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A method for the determination of pesticides that are registered for use in Canada on oilseeds such as canola, soybeans, mustard and flax using a modified QuEChERS approach has been developed. The Canadian Grain Commission is the leading Canadian authority on food safety issues affecting grains. Through monitoring, research and testing grain for toxic substances, the Canadian Grain Commission is able to show customers that Canadian grain is a safe commodity. With approximately 10 million tonnes of oilseeds being exported in the 2012/2013 crop year, the need for a dependable method of pesticide analysis was required. Canola contains >43% oil and has been an analytical challenge for trace level analysis of pesticide residues. A ground sample is extracted by shaking with an acetic acid/acetonitrile 1/99 (v/v) solution and MgSO4/NaOAc mixture. After centrifuging, a portion of the extract is cleaned up by a two-step cleanup using an MgSO4/PSA/C18E mixture. The sample is centrifuged in-between the two-stage cleanup and after the second cleanup; the extract is taken to dryness under nitrogen, made to a 1 mL volume in isooctane containing internal standard. 1µL is injected onto a GC-MS system and 1µL is injected onto a GC-ECD system.

P-18 Analysis of Mycotoxins in Cereals Using a Simple Extraction and LC-ESI/MS/MS with Fast Polarity Switching and Scheduled MRMs (Multiple Reaction Monitoring)

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A quick, sensitive and specific LC-MS/MS method has been developed for the detection of several major classes of known toxic and regulated mycotoxins. The method uses a simple solvent extraction followed by a further dilution and injection of sample extracts to achieve detection of mycotoxins below regulatory requirements. A Waters I-Class UPLC with a Phenomenex Kinetex 2.6µm XB-C18 column and integrated with an AB SCIEX Triple Quad 5500 using fast polarity switching and Scheduled MRMs to cover all the mycotoxins of interest. For the 25 mycotoxins analyzed, 17 compounds were detected in the positive ionization mode and 8 of them in the negative ionization mode. Quantitation was done using both C13 internal and matrix matched standards. Two precursor-product ion transitions quantify and confirm results.

P-19 How to maintain good peak shape of early eluting polar pesticides after the direct injection of QuEChERS acetonitrile extracts during LC-MS/MS analysis

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The pesticide residue laboratory at the Food and Environment Research Agency (Fera) has been using QuEChERS for the routine pesticide analysis of fruits and vegetables by LC-MS/MS since the beginning of 2004. Although the final acetonitrile extract is amendable to direct analysis by LC-MS/MS, the peak shape of early eluting polar analytes, such as acephate, asulam, methamidaphos and monocrotophos, is compromised. This distortion of peak shape leads to reduced sensitivity, poor precision and unsuccessful automatic integration. Peak shape of the polar analytes can be improved by better matching of the sample submission solvent with the initial mobile phase composition but this often results in precipitation of co-extractives and loss of certain analytes by hydrolysis, oxidation and binding to precipitate.

This study describes two approaches that allow the direct injection of acetonitrile extracts whilst maintaining good peak shape of the early eluting polar compound by facilitating better mixing during sample loading such that the analytes were subsequently dissolved in a more polar solvent when they entered the column, leading to isocratic band compression. These minor modifications require only limited re-validation but provide significant improvements in the quality of data for the polar analytes whilst avoiding the need and complications associated with adjusting sample submission solvent composition.

P-20 ESI/MS/MS Analysis of Neonicotinoid Insecticides in Canadian Prairie Agricultural Wetlands

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Neonicotinoids are systemic insecticides widely used in both North America and Europe primarily as a seed treatment for a diverse array of crops including the field crops that represent the bulk of agricultural production: canola (oilseed rape), cereals, soybean and corn. The environmental properties of this class of pesticides indicate they are persistent and have high leaching potential, and evidence in Europe and North America show that they are highly toxic to a wide range of non-target aquatic insects and other arthropods. The intensive and increasing use of these products in the Canadian prairies warrants a study to investigate the fate and effects of these chemicals in agricultural regions of the Canadian prairies – namely their impact on wetland ecosystems which may be contaminated through runoff. A robust sensitive analytical method using solid phase extraction and ESI/MS/MS was developed to detect and monitor four neonicotinoid insecticides (imidacloprid, thiamethoxam, clothianidin, and acetamiprid) in Prairie wetlands in order to determine their fate and effects in the environment. Neonicotiniod recoveries ranged from 66 – 77% (<10% RSD) in spiked river water. Data suggests that neonicotinoid insecticides are frequently found at detectable concentrations in Prairie agricultural wetlands.

P-21 Determination of Highly Polar Pesticide Residues in Food of Plant Origin, by an Automated QuPPe Solution

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The QuEChERS (Quick-Easy-Cheap-Effective-Rugged-Safe) sample extraction method was developed for the determination of pesticide residues in agricultural commodities. Since development, QuEChERS has been applied for the extraction of many different classes of pesticides, with one exception, very polar non-QuEChERS-amenable pesticides. In 2008 at EPRW in Berlin the "Quick Method for Analysis of Residues of numerous Highly Polar Pesticides in Food of Plant Origin involving Simultaneous Extraction with Methanol and LC-MS/MS Determination" (QuPPe-Method) was presented.
The aim of this project is to evaluate the performance and versatility of the AutoMate-Q40 for the extraction of highly polar pesticides using the QuPPe-Method. Liquid Chromatography coupled to a triple-quadrupole mass spectrometry (LC-MS/MS) was employed for the detection of highly polar pesticides 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 the QuPPE-Method this provides us with suitable analytical results falling in the method guidelines (range of 70-120% and RSD <20%) for the majority of the target compounds

P-22 Determination of Pesticide Residues in Honey, by an Automated QuEChERS Solution

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The QuEChERS (Quick-Easy-Cheap-Effective-Rugged-Safe) sample extraction method was developed for the determination of pesticide residues in agricultural commodities. While originally QuEChERS has been developed for plant matrices the technique has since been adapted to be used in several applications and in many additional matrices such as honey.

With the rise in popularity of this extraction technique and due to its reliable multiresidue methods it has driven the need for automation of the QuEChERS extraction to increase productivity and throughput. The AutoMate-Q40 streamlines the QuEChERS method from adding Acetonitrile (ACN) and buffering salts, shaking, mixing, centrifugating the sample, transferring to a dispersive solid phase extraction (d-SPE) tube, measuring and delivering the extract.

The aim of this project is to validate the extraction performance of the AutoMate-Q40 by monitoring neonicotinoids and other pesticides in honey. The target residues will be determined by Liquid Chromatography tandem mass spectrometry.

P-23 Determination of Atrazine residues in egg samples of *Podocnemis expansa* from the Brazilian Amazon

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The Brazilian agriculture becomes intense and the use of pesticides are necessary for have success in the harvest; due to this, some environmental compartments are contaminated by these compounds and their impact on the wildlife are not fully understood by the science. Atrazine is an herbicide largely used in Brazil and their endocrine disruptor properties and teratogenic effects are known by the science. Several researches showed their presence in water and soil. Some species of the order Testudines spawn in nests within or near agricultural areas and may be exposed to contamination by atrazine. To evaluate their effect, an experiment with authorization of the Center for Conservation and Management of Reptiles and Amphibians RAB/ICMBio Nº 35957-1/2012 and Ethics Committee on use of Animals of the Federal University of Uberlandia (CEUA/UFU 055/12) was installed; where eggs of *P. expansa* were collected from their natural ambience and artificially incubated in contaminated sand whit three levels of atrazine: 0.002, 0.02 and 0.2 mg kg^{-1}. For the evaluation of the presence of this compound in the content of the eggs, the QuEChERS method was validated using a LC-MS/MS system, a Zorbax Eclipse Plus C-18 3.0 x 100 mm 3.5 micron column to 30°C was used, the mobile phase were 30% acidified water with 0.1% of acid formic and 70% acidified acetonitrile with 0.1% acid formic, the flow was 0.6 mL min^{-1} the volume of injection was 20µL, nitrogen was used to 300°C the MS system was operated in positive mode with 4000 V, two transitions were used (216-173.9 and 216-103.9). The limit of quantification was 0.00015 mg kg^{-1}, two fortification levels were tested 0.01 and 0.1 mg kg^{-1} and the recoveries were 119 – 113% for each one, the CV were 3 – 5% and the uncertainty was 7%, a total of 32 samples were analyzed and not were founded atrazine residues.

P-24 Determination of Methyl Parathion residues in egg samples of *Podocnemis expansa* from the Brazilian Amazon

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Brazil in one of the most important agricultural producers in the world; the use of quality seeds, transgenic technologies, agricultural machines and agrochemical products are necessary for support this system. The insecticides are the second class of pesticides most used on the crops, Methyl Parathion is a most used insecticide in the world and in Brazil their used is permitted for rice, cotton, soybean, potatoes, onion, corn and wheat, some experiments showed that this compound can produce a reduction of the Ca-Mg-ATPase activity, with loss of calcium homeostasis in the cells and other reports changes in the skeletal development in quails, with shortening and twisting of the spine and defects in sternum, rib and tibia. No toxicity study for methyl parathion in reptiles was described. Some species of the order Testudines spawn in nests within or near agricultural areas and have high possibility to be affected by Methyl Parathion. To evaluate their effect, an experiment with authorization of the Center for Conservation and Management of Reptiles and Amphibians RAB/ICMBio Nº 35957-1/2012 and Ethics Committee on use of Animals of the Federal University of Uberlandia (CEUA/UFU 055/12) was installed; where eggs of *P. expansa* were collected from their natural ambience and artificially incubated in contaminated sand whit three levels of Methyl Parathion: 0.035, 0.35 and 3.5 mg kg^{-1}. For the evaluation of the presence of this compound in the content of the eggs, the QuEChERS method was validated using a LC-MS/MS system, a Zorbax Eclipse Plus C-18 3.0 x 100 mm 3.5 micron column to 30°C was used, the mobile phase were 30% acidified water with 0.1% of acid formic and 70% acidified acetonitrile with 0.1% acid formic, the flow was 0.6 mL min^{-1} the volume of injection was 20µL, nitrogen was used to 300°C the MS system was operated in positive mode with 4000 V, two transitions were used (264-232 and 264-125). The limit of quantification was 0.015 mg kg^{-1}, two fortification levels were tested 0.1 and 1 mg kg^{-1} and the recoveries were 119% for each one, the CV were 0.8– 0.7% and the uncertainty was 0.1%, a total of 32 samples were analyzed and not were found residues.

P-25 Remobilization of bound residues of herbicides in soils cultivated with sugar cane with vinasse application, straw and filter cake

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The system of production of sugarcane requires the intensive use of agricultural inputs. When applied in the culture of sugar cane these pesticides can follow different routes, reaching the soil directly or indirectly. In contact with the soil they can be absorbed by plant roots, leached, degraded or remain strongly sorbed on the soil. When a molecule of pesticide is sorbed to colloids, called bound residue, however, in some cases, part of the bound fraction to the soil can return to the soil solution by microbiological processes, and this process is known as remobilization. The application of vinasse to fertigation, straw and filter cake is widely used in the cane fields, but it is unclear whether this practice causes to remobilization and mineralization of bound residues and can bring benefits to the degradation of these herbicides by microorganisms, or affect the quality of the soil and can leaching to groundwater. This project evaluated, under laboratory conditions, the remobilization of bound residues of ^{14}C-diuron, ^{14}C-diuron + hexazinone, ^{14}C-hexazinone and ^{14}C-metribuzin in two soil cultivated with sugar cane, where were used known amounts of radiolabeled herbicides treatments with application of vinasse, straw and filter cake. Statistically, hexazinone was the herbicide that more mineralized in all treatments and in both soils. The best result for hexazinone was with straw for clay soil, and filter cake for sandy soil. The mixture had an antagonistic effect for hexazinone, in both soils; synergistic to diuron on sandy soil and antagonistic in clay soil.

P-26 Identification of metabolites of study remobilization of bound residues of herbicides in soils cultivated with sugar cane with vinasse application, straw and filter cake

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The environmental significance of the bound residues of herbicides presence and their metabolites in soil are important for new methods of herbicide application, it should be considered for these wastes transition following crops, their effect on the biota of terrestrial and aquatic systems, the potential transfer to the food chain, the potential for contamination of groundwater, and their long term effects on the quality and fertility of soils. Thus, the objective of this study was to identify the metabolites formed from remobilization and degradation of bound residues of 14C - diuron, 14C – hexazinone and 14C - metribuzin in two soils cultivated with sugarcane, with application of vinasse, filter cake and straw. Through analysis by thin layer chromatography (TLC), it was observed that there was formation of metabolites of herbicides and these were identified by ultra-efficiency liquid chromatography coupled to a mass spectrometer quadrupole time-of-flight. The analysis time was 9 minutes with column Acquity UPLC® BEH C-18 (1.7 µm, 2.1 mm d.i. x 100 mm) and 40ºC. Through the masses, molecular formulas and isotopic characteristics, was identified the metabolite DCPMU (1-(3,4-dichlorophenyl)-1-methylurea), belong to the diuron product, the metabolite B (3-cyclohexyl-6-(methylamino)-1-methyl-1,3,5-triazine-2,4-(1H,3H)-dione), belong to the hexazinone product, and the metabolites DA (Desamino-metribuzin), DK (Diketo-metribuzin), and DADK (Desamino-diketo-metribuzin), belong to the metribuzin product.

P-27 Evaluation of Environmentally Relevant Concentrations of Florfenicol on Fishes using the Comet Assay

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The use of antibiotic in Brazilian fish farming is a commonly activity for the improvement of the productivity. On the other hand, the intensive use contributes for its detection in aquatic environments causing toxicity to the biota. In Brazil, the antibiotic Florfenicol is used in fish farming, and as result of its use, is very important to verify how dangerous this compound is. The goal of this work was to evaluate the genotoxic potential of environmentally relevant concentrations of Florfenicol to tilapia (*Oreochromis niloticus*) using the comet assay. In a previous study at our laboratory, the concentration 480 µg L^{-1} was found in a Brazilian fish farming by LC/MS-MS, and in this study young of *O. niloticus* were exposed to this concentration, plus its half (240 µg L^{-1}) and the double (960 µg L^{-1}) during 96 hours with a repetition for treatment plus a negative and positive control. At the final of the test, blood from all fishes (n=12 per treatment) were collected by cardiac puncture, submitted to the comet assay in alkaline version (pH>13) under specific conditions for ADN denaturation, electrophoresis (running time: 20 min; Voltage: 39V and amperage 300 A) and then stained with GelRed. Comets were analyzed by visual methodology, considering the frequency of cells with or without damage, the score and the distribution of class. Kruskal-Wallis with $p<0.05$ was used to compare the ADN damages between the groups treated with antibiotics and controls. In conclusion, the higher concentration was genotoxic to the specie *Oreochromis niloticus*.

P-30 Validation of chromatographic method for Persistent Organic Pollutants (POP) pesticides in sediment from a recharge area of Guarani Aquifer

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Agriculture has been pointed as an important source of pollutants in the environment. Some dangerous chemicals used in agriculture (Mirex, Lindane, Endosulfan, BHC and etc.) were prohibited in the past six decades but can still be found at the environment in recent days due to the high environmental persistence. These chemicals are now under the scope of the Stockholm convention and presents phase-out dates to the end of use and final destination. The present work aimed to validate the chromatographic method for organochlorine molecules with POP's characteristics for GC-µECD analysis in sediments of aquifer area. Twelve substances were tested: α–BHC, β – BHC, Heptachlor, Dicofol, o.p, DDE, α–endosulfan, p.p, DDE, o.p, DDD, β–endosulfan, p.p, DDD, Endosulfan sulfate e Myrex. The extraction method for the validation was: 1g of sample, 5 mL of extraction solution (toluene+ethyl-acetate 1:1 acidified with 1% acetic acid), then vortex 1 minute, 10 minutes in centrifuge at 5000 RPM, collection and filtration of the supernatant and Injection of 1mL. GC-µECD was used with an HP 30mx320µmx0.25µm column, ChemStation software B.04.02, under the following conditions: oven temperature initial program of 100°C to 210°C (20°C/min) holding by 3 min and 210°C to 280°C (10°C/min) holding by 3 min; carrier gas (N), constant flow rate 1 mL/min; detector temperature 300°C; make up gas (N2) of 39 mL and total run of 22.8 min. The recoveries of fortification were between 70-120% (RSD<20%) at 3 spiking levels and 5 repetitions. All the analytical curves showed correlation coefficients greater than 0.999.

P-31 Determination of Atrazine and Methyl Parathion residues in substrate of nests of *Podocnemis expansa* from Brazilian Amazon

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The use of pesticides in an intensive agricultural production system like in Brazil, is a necessity; this country is one of the most important consumers of pesticides in the world, pesticides such as Atrazine that is an herbicide largely used on the sugar cane production area and their endocrine disruptor properties and teratogenic effects are known by the science; Methyl Parathion can produce a reduction of the Ca-Mg-ATPase activity, with loss of calcium homeostasis in the cells and other reports changes in the skeletal development in quails, with shortening and twisting of the spine and defects in sternum, rib and tibia. Some substrate of the nests of the species of the order Testudines are near of agricultural areas and have high possibility to be affected by Atrazine and Methyl Parathion. By these reasons, the QuEChERS method without clean-up step was validated using a LC-MS/MS system, a Zorbax Eclipse Plus C-18 3.0 x 100 mm 3.5 micron column to 30°C was used, the mobile phase were 30% acidified water with 0.1% of acid formic and 70% acidified acetonitrile with 0.1% acid formic, the flow was 0.6 mL min^{-1} the volume of injection was 20µL, nitrogen was used to 300°C the MS system was operated in positive mode with 4000 V, for Atrazine detection two transitions were used (216-173.9 and 216-103.9) with a limit of quantification of 0.00015 mg kg^{-1} and for Methyl Parathion (264-232 and 264-125) with a limit of quantification of 0.015 mg kg^{-1}, for the recovery test, two fortifications levels were used 0.01 – 0.1 mg kg^{-1} for Atrazine and 0.1 – 1 mg kg^{-1} for Methyl Parathion the recoveries were near from 118 % for both compounds and CV >7% the uncertainty >13%. The method is adequate for determination of Atrazine and Methyl Parathion in substrate of nest of Testudines.

P-32 Presence of PAHs in sea algae samples from a marine protected area "Fernando de Noronha in Brazil

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The presence of polycyclic aromatic hydrocarbons (PAHs) in marine compartments were reported by several researches in the world, and its impact on the populations that habit these ecosystems are not fully understood, Due to its hydrophobicity, the PAHs are preferentially deposited on the sediments and the sediments are an important source of nutrients for algae growing in these environments and can be absorbed by the algae by these reason the contaminated sediments are an important dissipaters of PAHs in the aquatic environment, There is the suspected of binding of these contamination with the development of neoplasm in some animal, for example the fibropapillomatosis in species of sea turtles, problem that is endangering both threatened and endangered marines species. Fernando de Noronha that is an important area for feeding and reproduction of sea turtle; by these reason 21 samples of sea algae were analyzed for determinate the presence of 15 PHAs (naphthalene acenaphthene, anthracene, benz[*a*]anthracene, benzo[*a*]pyrene, benzo[*b*]fluoranthene, benzo[*ghi*]perylene, benzo[*k*]fluoranthene, chrysene, dibenz(a,h) anthracene,fluoranthene, fluorene, indeno(1,2,3-cd)pyrene, phenanthrene, pyrene). A liquid chromatography system coupled to fluorescence detector were used for the detection and quantification of these compounds; the method showed linearity of ≥ r^2 0,99 and not matrix effect, limit of quantification in order to 0,0006 to 0.07 mg kg-1; recoveries between 67,9 – 165,4 %. From the 21 sediments samples analyzed, all showed contaminations by naphthalene and the maximum concentration founded were 0.16 mg kg-1. These results showed contamination by these compounds in areas without a high presence of the human population, because Fernando de Noronha is a marine protected area, these concentration can be harmful for the population of the animals who life on these area.

P-34 Monitoring of PAHs residues in sediments from the Iner Bay of the Titicaca Lake in Puno – Perú

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The Titicaca Lake lies in the South American Andes at the border of Peru and Bolivia, with average altitude of 3810 meters above sea level, is considered as the highest navigable lake around the world, it´s covers 8400 km2, has approximately 932 km3 and constitutes the freshwater source for nearly three million people; the inner bay represent the 2.1% of the lake, is one of the most vulnerable areas, this area is affected by the organic compounds such as pesticides and bacteriological contamination caused by direct discharges of wastewater from the urbanized areas of Puno city and runoff from crop areas around the lake. The presence of organic contaminants in aquatic compartments such as polycyclic aromatic hydrocarbons (PAHs) were reported by several researches in the world, and its impact on the populations that habit these ecosystems are not fully understood, Due to its hydrophobicity, the

PAHs are preferentially deposited on the sediments of the aquatic ecosystems and the contaminated sediments are an important dissipaters of PAHs in the aquatic environment; by these reason 13 samples of sediments from the Titicaca Lake were analyzed for determinate the presence of 15 PHAs (naphthalene acenaphthene, anthracene, benz[*a*]anthracene, benzo[*a*]pyrene, benzo[*b*] fluoranthene, benzo[*ghi*]perylene, benzo[*k*]fluoranthene, chrysene, dibenz(a,h)anthracene,fluoranthene, fluorene, indeno(1,2,3-cd) pyrene, phenanthrene, pyrene). A liquid chromatography system coupled to fluorescence detector were used for the detection and quantification of these compounds; the method showed linearity of ≥ r^2 0,99 and not matrix effect, limit of quantification in order to 0,0005 to 0.08 mg $kg^{-}1$; recoveries between 79 – 121 %. From the 13 sediments samples analyzed, 11 showed contaminations by the less one of the 15 PAHs analyzed, one samples showed contamination by ten PAHs, and the maximum concentration founded were 0.57 mg $kg^{-}1$ of benz[*a*]anthracene. These results showed contamination by these compounds, these concentrations can be harmful for the population of the animals who life on this area and it can be a risk for the human population of this city because the water of this lake is used as drinking water.

P-35 A simple and low-cost method for analyzing multiple veterinary drug residues in foods of animal origin in Vietnam

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The emergence of multi-drug resistant bacteria (MDRB) is thought to have been driven by excessive use of antibiotics in the livestock and fishery industries, as well as in healthcare settings. Vietnam has a higher incidence of MDRB compared with industrialized countries, such as Japan, likely due to antibiotic abuse on farms. To assess the extent of antibiotic contamination of food in Vietnam, a simple and practicable method was needed. We developed the following low-cost method that targeted a wide-range of antibiotics such as sulfamides, quinolones and beta-lactams. A homogenized meat sample aliquot (2 g) was weighed in a centrifuge tube and mixed with 10 mL of 80% acetonitrile using a Polytron® homogenizer for 1 min followed by centrifugation at 2000 x *g* for 3 min. The extract (upper layer) was decanted into a dispersive-solid phase extraction tube containing 0.3 g ODS particles, shaken for 1 min, and centrifuged at 2000 x *g* for 3 min. An aliquot (1 mL) of the upper layer was transferred into another tube containing 1 mL of distilled water. The mixture was filtered by PTFE membrane before LC-MS/MS analysis.
We analyzed over 400 samples including poultry, swine, cattle, shrimp and fish collected in Ho Chi Minh City and NhaTrang in 2013 - 2014. Three compounds were detected frequently, sulfaclozine from poultry, sulfadimidine from swine, and enrofloxacin from poultry and fish. High concentrations (over 1 µg/g) of sulfamides were detected in four samples. Overall, antibiotics were detected in about 10% of the samples collected in Vietnam.

P-36 Development and Validation of an Analysis Method for the Determination of Glucuronolactone in Beverages using the LC-MS/MS Triple Quadrupole Mass Spectrometer

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Glucuronolacrone is a chemical compound mainly used in energy drinks with claims that it detoxifies the body. The use of glucuronolactone as a medicine is permitted in Korea but as food additives in beverages is prohibited. To prevent illegal use of glucuronolactone in beverages, glucuronolactone in beverages has been analyzed consistently. Due to its high polarity, low molecular weight and absence of chromophores, glucuronolactone is analyzed in LC-PDA chromatography through derivatization by phenyl methyl pyrazolone. Besides complex analysis steps, other saccharides in beverage can cause false positive results in LC-PDA analysis method.

In this study to improve the drawbacks of conventional method, an accurate, simple, reproducible and sensitive method for the determination of glucuronolactone has been developed using LC-MS/MS triple quadrupole mass spectrometer. To retain glucuronolactone in column, Hydrophilic Interaction chromatography [HILIC] technique is adopted using amide column and acetonitrile rich mobile phase. The developed method was fully validated with respect to selectivity, linearity, limit of detection, limit of quantification, precision, accuracy and repeatability/reproducibility. All validation parameters were acceptable to AOAC Guidance for Standard Method. With developed method, 9 energy drinks sold in Korea were analyzed and glucuronolactone was not detected in all samples.

P-37 The Q&A Handbook for Pesticide Residue Analysis by the Pesticide Society of Japan: English Version Progress Report

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At the 2013 NACRW the authors introduced a convenient, simple, Q&A format handbook that is dedicated to the area of pesticide

residue analysis published by the Pesticide Society of Japan. The need for easy to use, up to date, and practical references in the area of the pesticide residue analysis was communicated to the audience. Encouraged by the support from the workshop participants the authors decided to translate the whole handbook into English. The translation is almost complete. The handbook consists of about 100 questions with each answer succinctly summarized in less than 2 pages by the experts of each category. It can be used as a reference to read only the pages that are related to the questions one is facing or it can be read through for a quick review of the pesticide analysis work. Topics covered include: regulatory standards, analytical apparatus, sampling, extraction and cleanups, instrumental analysis, quality control, and residues in environment. The categories/questions that received most requests for translation are revealed in the poster. Through the presentation the authors hope to discuss with the audience the most effective way of distributing the English version to the pesticide residue society.

P-38 Analysis of emerging contaminants by GC x GC combined with high-resolution mass spectrometry: using exact mass information to explore the data.

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Comprehensive two-dimensional gas chromatography (GC x GC) in combination with high-resolution mass spectrometry (HRMS) is a powerful tool for the analysis of complex mixtures. However, new software tools are required to facilitate the interpretation of the rich information content in GC x GC/HRMS data sets.

In this work, we analyzed a dust sample collected from an electronics recycling facility by using GC x GC in combination with a new high-resolution time-of-flight (TOF) mass spectrometer. A composite mass spectrum was created by summing the mass spectra for all components in the GC x GC/HRTOFMS analysis. Halogenated contaminants were readily recognized by their mass defects. The mass defect plots facilitated rapid identification of families of compounds that differ by the number of chlorine and bromine substituents. This approach also helped guide the analysis of the chromatographic data.

Mass chromatograms were generated based on specific ions identified in the plots as well as regions of the plots predominantly occupied by halogenated contaminants. Tentative identification of specific contaminants was aided by database searches and elemental composition determinations from the exact-mass data. Software tools that incorporate nontraditional Kendrick mass defect plots greatly enhanced the interpretation of the GC x GCHRTOFMS data. The details of these results will be reported for this presentation.

P-39 Statistical Determination of Stability from Two Multi-Component Pesticide Mixes

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A study was conducted to determine the stability of large “mega” mixes containing pesticides, metabolites, and contaminants. The evaluation consisted of two mixes, each comprised of 323 LC-amenable and 210 GC-amenable components. Both 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. These compounds covered a broad spectrum of physical-chemical characteristics. Conversely, 143 compounds (twenty-seven percent) across the two mixes showed statistically relevant degradation between the control and six-month heat treatment. Numerous herbicides within specific chemical classes (urea, phenylurea, cyclohexene oxime, and carbanilates) showed measureable degradation across all analytes within each class. Additional compounds across many other chemical classes also degraded; however many of these compounds contained chemical features unique to the compound and not representative across the chemical class. A small selection of compounds exhibited enhanced response between the control and heat treatments. The proposed explanation for this increase involved the breakdown of larger precursor pesticides into the smaller compounds, also present in the mixes. Most notably, select analytes showed excessive breakdown between the control and six month heat treatment; this provided key insight into the importance of preparing those compounds within the laboratory, rather than including them within a standard mix of sufficient size.

P-40 New Column Technologies for GPC Cleanup in Aquatic Tissue Matrices

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The current EPA Gel Permeation Chromatography (GPC) Cleanup procedure (Method 3640A) recommends a maximum lipid load for a 5mL injection onto the column of 1 gram. This limit often necessitates multiple aliquots of the same sample to be processed separately in order to remove the lipid while adequately cleaning the sample prior to analysis. This requires the use of more solvent, increasing the overall cost and labor of sample cleanup. Furthermore, many laboratories are under pressure to decrease solvent usage. While alternatives are available to reduce solvent, it often means a reduction in lipid capacity, eliminating any benefit. In this study we will use PrepLinc™ GPC Maxx to increase lipid load while minimizing solvent usage and runtime. GPC Maxx allows the user to control the standard GPC Dump, Collect and Wash operation through two columns in tandem. The lipid capacity of each sample is increased by reducing the lipid load onto the second column and maximizing lipid removal in a single sample injection. We will demonstrate the effectiveness of GPC Maxx in the cleanup of different types of aquatic tissue for the analysis of polychlorinated biphenyls and organochlorine pesticides in comparison to the traditional GPC Cleanup technique. We will also demonstrate the usefulness of alternative column technologies to further increase lipid removal while minimizing overall runtime.

P-41 A Technique for the Simultaneous Analysis of Pesticide Residues in Multiple Agricultural Commodities using Matrix Replacement

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Laboratories wage a constant battle against matrix effects when using tandem mass spectrometry (GC-MS/MS and LC-MS/MS) for pesticide residue analysis in food and agricultural commodities The most common techniques for minimizing matrix suppression and enhancement are sample extract cleanups (d-SPE and SPE), dilution, and matrix matched calibration standards. Additional cleanup steps can affect the time and cost of residue analysis. Insufficient MS/MS sensitivity may limit the dilution factor of sample extracts prior to analysis. Matrix matched calibration standards work well when analyzing samples of the same commodity, but do not allow for the concurrent analysis of multiple commodity types in a single analytical sequence.

Matrix replacement is a technique in which a reference matrix is added to all calibration standards and sample extracts at the same concentration. The reference matrix exhibits “stronger” matrix effects than the native matrix of the samples. Combined with a dilution factor (5-10x), the reference matrix becomes the dominant matrix for both the calibration standards and samples, allowing extracts of different commodity types to be quantified using a single calibration curve.

Samples of three different commodities were spiked and extracted using a QuEChERS procedure, modified with reference matrix and analyzed at various dilution factors to determine the point where the reference matrix normalizes the response for each individual pesticide. Spiked commodities were analyzed using matrix matched and matrix replaced calibration curves to compare the accuracy and precision of the two techniques.

P-42 Multiresidue pesticide analysis of dried botanical dietary ingredients according to USP561 using QuEChERS and GC-Triple Quatrupole Mass Spectrometry

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Pesticide residue analysis of plant based dry dietary ingredients used in dietary supplements is among the most difficult to perform due to major matrix interferences. Multiple pesticide residue methods have been published; however, only few were evaluated on material of botanical origin.

A simple and robust multiresidue screening method based on the USP561 analyte list was developed and validated for the analysis of pesticides in 6 representative dry botanical dietary ingredients. We used a QuEChERS extraction method with a modified dispersive SPE coupled with GC-MS/MS determination. Matrix-matched standard calibration curves showed linearity R2 > 0.990 across a concentration range of 0.2-500 ng/mL for the majority of the 86 pesticides evaluated in the botanical matrices. Accuracy and precision were evaluated through fortifications of all 6 botanical ingredients at 10, 25, 100, and 500 µg/kg. Triphenyl phosphate was used as an internal standard to control for analyte recovery and matrix effect. Mean pesticide recoveries and relative standard deviations (RSDs) for all botanical ingredients were 96%, 92%, 95%, and 90% at 12%, 8%, 15%, and 10% RSD for 10, 25, 100 and 500 µg/kg respectively. Validation of the representative botanicals showed applicability of the method for routine screening of USP561 specified pesticides in a wide variety of botanical dietary ingredients.

P-43 Florida’s Pesticide Residue Regulatory Program – FY 13 - 14

Jo Marie Cook

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Each fiscal year, from July to June, Florida's Chemical Residue Laboratory screens hundreds of fresh fruits and vegetables for pesticide residues. The multi-residue screen has been expanded to over 300 analytes with many detection limits at 10 ppb. Honey has also been added to the screen. Florida's pesticide regulatory program focuses on home grown products and commodities that have been found violative in the past. This fiscal year also marks the end of a 3- year program to screen seafood for PAH's and dispersant. The poster will summarize analytical findings and detail violations by commodity and pesticide. Commodities with the most pesticides in a single sample and the frequency of pesticides by commodity will be shown. Overall, the incidence of violations remains very low, even in a program designed to target problems.

P-44 Modified QuEChERS Multi-Residue Analysis of Neonicotinoids in Honeycomb Using Orbitrap Technology

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With the growing concerns of Citrus Greening, neonicotinoids have taken a prominent use in the citrus industry to control the vector, Asian Citrus Psyllid. Due to increased use, the capability to monitor these compounds is critical. A citrate buffered QuEChERS extraction was modified to more efficiently extract pesticides from wax. Honeycomb samples were homogenized with dry ice prior to extraction. Three grams of honeycomb were dispersed into the water phase to release any compounds trapped inside the wax using a 65°C water bath, prior to the addition of the acetonitrile and citrate buffer. After the sample is extracted with acetonitrile, it is treated with a -20°C freeze out step to assist in the mitigation of the wax. In this work, honeycomb was spiked with 7 neonicotinoids including a metabolite of Imidacloprid. Analysis was performed on a Thermo Exactive Plus Orbitrap LC-MS. Simultaneous full scan and All-Ion-Fragmentation with high energy collision dissociation scans were utilized at a resolving power of 70,000. Data sets from April 2013 through April 2014 were compiled to evaluate method performance.

P-45 Method Validation for a modified QuEChERS approach to quantify 185 pesticide residues in fresh salmon by LC-MSMS and GC-QQQ

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The application of the quick easy cheap effective rugged and safe (QuEChERS) method has been widely used in the preparation of fruit and vegetable commodities (e.g. lettuce, mushrooms, and apple juice) for pesticide residue analysis. In this study, we successfully applied a modified version of the QuEChERS extraction method to quantify 185 pesticide residues in fresh salmon samples. Analysis of these compounds was performed using a combination of LC-MSMS and GC-QQQ instrumentation. The validated QuECHERS method uses ethyl acetate for the extraction solvent and involves two freezing steps for removal of lipids. The LC-MSMS provided high sample throughput with 15 minutes analysis time for 104 pesticide residues and the GC-QQQ provided medium sample throughput with 45 minutes analysis time for 81 pesticide residues. In a quantitative validation, acceptable performances were achieved with overall recoveries of 70-120% and < 20% RSD for 149 analytes in 7 sample extracts over the course of 5 different extractions at the two times the limit of quantitation spiking level. Over the course of 12 months, this method will be tested on 700 salmon samples as part of the U.S. Department of Agriculture, Pesticide Data Program; samples include wild and farmed, imported and domestic, skin and skinless, pacific and atlantic for the following species: Chinook, Chum, Coho, Pink, and Sockeye.

Mention of brand or firm name does not constitute an endorsement by the U.S. or Washington State Department of Agriculture above others of a similar nature not mentioned.

P-46 Method modifications to FDA method LIB4306 allow for the determination of chloramphenicol residues in food grade enzyme powders by SPE-LC-ESI-MS/MS

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The fermentation process associated with the manufacture of food grade enzyme powders was recently determined to support the colonization of bacteria. To control this issue, manufacturers following different international regulatory procedures have found the addition of familiar antibiotic compounds to manufacturing processes beneficial. This becomes an issue of public health as the concentration of antibiotic residues in food grade materials promotes unknown exposure to the global community. Continued interest in the biomonitoring of these compounds have inspired a number of method development strategies; however, classic methods are labor intensive and require multi-step, time-consuming efforts. The FDA has recently mandated a method for the determination of chloramphenicol in shellfish. This study evaluates the feasibility of method modifications to incorporate enzyme powder matrices into this method. Linearity was determined over a range of 0.1 – 5 ppb ($r^2 > 0.990$). Relative recovery was determined >80%. The typical repeatability (%RSD) for n=7 replicates <20%. Strategies to control solids prior to SPE load will be presented, as they were determined critical in method performance. Method robustness was determined by evaluating multiple

lots and multiple variants of enzyme powder origin. The key variable is the nature of the excipients as high level sugars of different sources need to be controlled to minimize the ESI suppression.

P-47 Quantitative Analysis of Endocrine Disrupting Compounds at ppt levels in Consumer Composts and Soils using HRGC/HRMS

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The organic food industry has grown considerably along with the boom of home-grown produce. Consumers often choose organic practices because they desire to lower their exposure to pesticides and other man-made residues found on commercial produce. Soil amendments that are labeled "organic" may not be free of endocrine disrupting compounds including chlorinated pesticides, chlorinated dioxins and furans and PCBs. This poster looks at the presence of these compounds in commercial organic compost, organic potting soil, organic garden soil and homemade compost. The exclusive use of HRGC/HRMS coupled with isotope dilution allowed for quantitation of analytes down to sub ppt levels. Unique clean-up strategies were employed for these complex matrices to improve both baseline and peak shape required for low-level analyses.

P-48 Examination of Pesticides in Wine, Beer and their Constituent Products using High-Throughput Techniques to Maximize Extraction & Efficiency

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There are hundreds of commercial pesticides in use in industrial and private agriculture. The concern over human pesticide exposure over the past few decades has led to the monitoring of these pesticides. Increasing concern over the health effects of residual pesticides on fruits and vegetables has led to increased testing of these products to determine the levels of pesticides on produce when it goes to market.

In this study, commercial red wine and beer samples were examined for their pesticide concentrations. In addition to the examination of the finished alcoholic beverage, the constituent agriculture products of wine a beer production: grains, malts hops and wine grapes; were also examined to determine the levels of pesticides found in those products. The sample preparation and extraction process efficiency and recovery were examined by processing samples using manual versus high-throughput techniques. The QuEChERS method was used to process a greater number of samples in a shorter period of time than other extraction methods.

P-49 More Efficient US EPA Method 8081with SPE Extraction of Organochlorine Pesticides

Michael Ebitson, Horizon Technology, Salem, NH, USA

Pesticide pollution is a subject of global concern, and although many countries have now banned the use of organochlorine pesticides, they linger in the environment and can contaminate water sources. Pesticides are toxic to animals and humans, so methods which accurately and easily quantify them are essential to the future.
The purpose of this work is to establish a solid phase extraction (SPE) procedure to streamline the sample preparation process for determining organochlorine pesticides from water.

The goals of the method are to:

- Use much less solvent
- Avoid emulsion formation
- Minimize possible contamination
- Eliminate the need for solvent exchange before the chromatographic step

Achieving these goals while meeting all of the QC requirements, including excellent spike recoveries, will be demonstrated.

P-50 Determination of Monocrotophos, Diazinon, Malathion, EPN, and Methamidaphos from Aqueous Samples Using Atlantic HLB SPE Disks

Jim Fenster, Julie McGettrick , Zoe Grosser, Horizon Technology, Inc., Salem, NH, USA
Methamidaphos, Monocrotophos, Diazinon, Malathion, and EPN are commonly used organophosphate pesticides for the control of insects and aquatic pests in rice production, other agricultural production, and fish aquaculture in parts of the world.

Methamidaphos in particular is used in great quantities in rice fields in China where rice–fish culture is common as well as in many other rice-producing countries (e.g., Thailand, Malaysia, and the Philippines).
Given their prevalent use throughout Asia, residues of Monocrotophos, Diazinon, Malathion, EPN, and Methamidaphos are detected

in many food sources and are commonly monitored in wastewater and drinking water in these regions. It would be more efficient to extract these pesticides in one procedure, rather than two, saving time and reducing cost. This poster will describe method development for automated extraction of this suite of polar pesticides and discuss the possibility of using the same concepts for other polar pesticides.

P-51 Identifying Unknown Chemicals and Disinfection Byproducts in Swimming Pools and Hot Tubs

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Swimming pools are treated with disinfectants to protect swimmers from pathogens and prevent illness. The water used to fill a swimming pool if from a municipal drinking water supply, is also often treated with disinfectants such as chlorine. Disinfectants will react with naturally occurring organic matter in water and, in the case of swimming pools, they can also react with chemicals introduced to the water by the swimmers themselves to produce byproducts that can be potentially harmful. It is important to treat water while minimizing the risk of disinfection byproducts (DBPs). One of the first steps is to chemically characterize the DBPs in swimming pools and hot tubs, very complex matrices, using non-targeted analysis since a lot of the contaminants are unknowns. Comprehensive two-dimensional gas chromatography time-of-flight mass spectrometry (GC×GC-TOFMS), as well as, GC high resolution TOFMS (GC-HR-TOFMS) were used for the identification of "known unknowns" and "unknown unknowns" in swimming pool and hot tub water. The "known unknowns" were identified by library database searching deconvoluted spectra using Leco's Chromatof software, while the "unknown unknowns" were tentatively identified using a combination of EI and CI accurate mass data.

P-52 A Novel Approach for the Post-Targeted Analysis of POPs by GC-HR-TOFMS

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The environmental analysis of persistent organic pollutants (POPs) in complex matrices often yield additional unknown peaks in a chromatogram beyond the target list, which begs the question, what else is in my sample? A unique attribute an analyst can use to help address this question is mass defect, the difference between the nominal and exact mass of a compound or chemical element. The use of mass defect in mass spectrometry has increased in recent years as mass analyzers become increasingly more sensitive and selective. Modern advances in time-of-flight mass spectrometry (TOF MS) have enabled them to achieve more than 25 k resolving power routinely, while acquiring full mass range spectra suitable for non-targeted analysis. In this study, whole fish extracts were cleaned up using gel permeation chromatography only, and analyzed using an Agilent multi-mode inlet and 7890 GC coupled to a Leco Pegasus HRT, high resolution time-of-flight mass spectrometer, operated in EI mode. The accurate mass data was scaled for hydrogen substitution of chlorine (Cl-H) by a factor of 34/33.96102 for the ratio between the nominal and exact mass of Cl-H. Plots of Cl-H Scaled Mass Defect versus Nominal (Cl-H) Scaled Mass were used to identify halogenated compounds in the samples. The most prevalent features corresponded to legacy compounds such as polychlorinated biphenyls, organochlorine pesticides (i.e., DDT metabolites), and polybrominated diphenyl ethers (PBDEs). Although, other halogenated compounds were identified in these samples that were formerly unknown, including polychlorinated styrenes, methoxy-PBDEs, as well as pentabromobenzene and other novel flame retardants.

P-53 Analysis of Mycotoxins Using a Mixed-Mode Solid-Phase Extraction Method and LC-MS/MS Detection Using a Polyaromatic HPLC Column

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Mycotoxins are toxic natural secondary metabolites produced by several species of fungi on agricultural commodities in the field or during storage. To date, more than 300 mycotoxins have been identified in cereals and other agricultural commodities and possessing varying degrees of toxicity. Mycotoxins are chemically stable and cannot be destroyed during food processing and heat treatment, thus, monitoring of these compounds in food is an important health, agricultural production, food processing and trade concern. The analysis of mycotoxins is challenging due to the large number of compounds to be detected and the wide physicochemical properties they possess. In addition, the food commodities tested are typically complex in nature and are often simultaneously contaminated with several mycotoxins at low concentrations. Sample preparation approaches reported for mycotoxin analysis include solid–liquid extraction, liquid–liquid extraction, matrix solid-phase dispersion, QuEChERS, immunoaffinity chromatography and solid-phase extraction (SPE). All approaches, including SPE, are complicated by the considerably different polarity and solubility of the mycotoxins, in particular the polar trichothecenes. Due to the limited wash step(s) that can be incorporated into an SPE method (due to loss of analytes), final sample extracts may still contain a large amount of matrix components that can affect the analytical detection system. Liquid chromatography coupled with tandem mass spectrometry (LC-MS/MS) has become the most universal detection system for mycotoxin analysis. However, challenges still remain, including the retention of the very polar trichothecenes.

To overcome some of the limitations of existing methods, there was a need to develop an extraction and clean-up method for the simultaneous determination of several mycotoxins with high recoveries of the polar toxins and minimizing the sample matrix effects. This poster details an optimized method for the extraction and clean-up of mycotoxins from grain-based food using a mixed-mode polymeric SPE sorbent. After solvent extraction, the mycotoxins were retained, rinsed and eluted from the neutral functionality of the SPE sorbent. The ion-exchange functionality retained charged matrix components that would otherwise co-elute into the final sample extract and affect the detection system. LC-MS/MS was used for the accurate detection and quantification of the mycotoxins. HPLC separation was successfully conducted using a Selectra® DA column, a polyaromatic sorbent which is capable of greater retention of the polar trichothecenes compared to a C18 stationary phase. The compounds included in this method are representative of a wide range of mycotoxins, including type A- and B-trichothecenes, aflatoxins (B1, B2, G1, G2), ochratoxin A, alternariol, zearalenone, zearalonol (α-, β-) and fumonisins (B1, B2).

P-54 New Sample Preparation Methodology to enable Higher Recovery, and minimize loss of difficult Analytes in Pesticide and Fungicide Panels 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 (which need to be concentrated). Several studies comparing these techniques with eXtreme filter vials (patented) for contaminant analysis were conducted in orange juice, soil, milk, shellfish and water analysis.

P-55 Are Fatty Acids Overwhelming your Dispersive SPE Cleanup and Causing Issues in your GC Analysis? Get More Cleanup Capacity with a Fast Sample Pass Through on a PSA Solid Phase Extraction Cartridge.

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Dispersive solid phase extraction (dSPE) is an important part of the QuEChERS sample preparation approach, as it allows a fast cleanup of extracts by shaking with loose sorbent material. PSA (primary secondary amine) sorbent is used in the dSPE cleanup to remove fatty acids. Many sources of dietary fatty acids are in fruits, vegetables, seeds, nuts, and animal fats. Removal of fatty acids prior to analysis is important as they can interfere with target analytes, making trace level determination difficult. GC injection port and column performance also quickly degrade when fatty acids are still prevalent in the sample extract.

In some high fat matrices the capacity of the dSPE format (50 mg / 1 mL extract) is just not enough to provide an effective cleanup of the extract for analysis. Increasing cleanup capacity by addition of more sorbent into the centrifuge tube is not practical because more of the sample is lost to the procedure. This also marginalizes a concentration step for improving GC detectability if needed. A more traditional cartridge solid phase extraction (cSPE) cleanup facilitates the use of larger amounts of sorbent (500 mg) while recovering all of the sample volume. We used a 500 mg PSA cartridge to remove fatty acids in complex matrices. The sample was simply loaded onto the cartridge and pulled through with vacuum. No elution solvents or sample concentration steps were necessary.

P-56 Modified QuEChERS and Shoot-and-Dilute GC: Fast Sample Preparation and Analysis of Brominated Flame Retardants in Fish

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Sample preparation is often the bottleneck in the analytical laboratory. Soxhlet or pressurized liquid extractions (PLE) are commonly used and can take several hours and hundreds of milliliters of solvents. Lipophilic compounds like brominated flame retardants also require extensive sample cleanup to remove fat from the matrix prior to injection on the GC. Utilizing the QuEChERS sample preparation concept and minimal extract cleanup can reduce solvent consumption to less than 50 mL and cut overall sample prep to a few hours. Relying on the sensitivity of the electron capture detector (ECD) for multiply halogenated compounds, we can perform a split injection. This Shoot-and-Dilute technique is analogous to Dilute-and-Shoot for LC-MS/MS analysis. Shoot-and-Dilute GC-μECD (split injection) is advantageous over typical splitless injections because it decreases the residence time of thermally labile compounds, like BDE 209, in the hot injection port and increases system uptime by depositing less nonvolatile material onto the column. Pairing the fast sample preparation concept of a modified QuEChERS and Shoot-and-Dilute GC, we evaluated several fatty food matrices for both newer flame retardants found in Firemaster® 550 and historical polybrominated diphenyl ethers (PBDEs).

P-57 Wool Packing or No Wool Packing in a Splitless GC Inlet Liner – What is Better for Pesticide Analysis? A Case Study with a QuEChERS Strawberry Extract

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Gas chromatographers analyzing pesticides typically avoid wool-packed inlet liners for splitless injection for multiple reasons, including irreversible sorptive loss of lower levels of active pesticides, thermal degradation of sensitive pesticides, and poor transfer of lower volatility pesticides to the GC column. But a wool-packed liner can provide better sample homogenization, resulting in more repeatable and accurate data. Wool also protects the GC column from non-volatile "dirt". Properly deactivated wool does not necessarily lead to loss of active pesticides, even at low levels, and a judicious choice of purge valve times permits complete transfer of relatively involatile pesticides.

In the work presented here, a selection of organophosphorus, organochlorine, organonitrogen, carbamate, and pyrethroid pesticides in a QuEChERS extract of strawberries was analyzed using hot splitless injection GC-MS for quartz wool-packed, borosilicate wool-packed, and empty single-taper inlet liners. Liners and wool were thoroughly deactivated to minimize losses of pesticides due to activity. Average response factors were plotted for 60 back-to-back injections for each liner type.

P-58 An Update on the QuEChERS Tablet

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We invented a QuEChERS salts tablet, which made the QuEChERS extraction method for pesticides in fruits and vegetables even easier as regards dispensing the phase-separation salts. However, difficulties in making the tablet inhibited its production. Recent breakthroughs in the manufacturing process on a bench scale have enabled a better performing tablet that is easily handled and yields quantitative results comparable to commercially-available QuEChERS salt packets. This presentation will share some promising results from this research.

P-59 Comparison of Two-Dimensional Gas Chromatography Time-of-Flight Mass Spectrometry and Gas Chromatography Tandem Mass Spectrometry for Pesticide Analysis in Herbal Teas

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Herbal teas are formulated using blends of herbs, plants, and spices. As with any plant-based commodity, there is high potential for pesticide residues to remain in the final product. Dried plant material found in herbal tea poses a significant analytical challenge with respect to pesticide analysis. The extract, even after an extensive cleanup, can contain a large amount of coextractive material that can overwhelm target pesticides, making trace detection very difficult.

In order to assess the impact of matrix complexity, several types of tea that varied in composition were used. Pesticides varying in polarity, volatility and pH stability were tested. Several strategies to minimize matrix interferences were employed. A modified QuEChERS method with a reduced sample size of 1 gram was used.

GCxGC-TOFMS was used to chromatographically separate matrix components from target pesticides because matrix interferences and recovery determination proved too difficult with 1D GC-TOFMS. Employing GCxGC-TOFMS also allowed use of the less intensive dSPE procedure without additional cleanup steps. A deactivated guard column (retention gap) was used prior to the first analytical column to increase system ruggedness and improve early eluting analyte peak shapes, resulting in improved detectability. The GC-MS/MS method afforded increased sensitivity for most target analytes. Three SRM transitions were monitored for each analyte increasing selectivity and identification confidence. Still, there were instances of matrix corruption of one or more transitions for several pesticides. The percent recoveries determined using both techniques were comparable but with differences in overall sensitivity and degree of matrix interferences.

P-60 Shoot-and-Dilute GC: Feasibility of Split Injection when Paired with Very Sensitive Detectors

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Shoot-and-Dilute GC (aka "split injection") is an analogous technique to dilute-and-shoot LC-MS/MS which is designed to mitigate matrix effects for pesticide residue analysis. Split injection GC is often used with concentrated samples but can be intentionally employed to alleviate matrix related issues occurring at the inlet and column.

There are well-known problems associated with splitless injection of dirty samples, most notably compound degradation and drastic response changes observed as the GC inlet liner becomes dirty. This can occur very quickly with real samples, sometimes with a single injection of a particularly dirty sample. Inlet and column maintenance are needed to restore instrument performance resulting in instrumental down time.

An easy way to diminish the aforementioned problems is to use split injection GC when possible. That is if LOD and LOQ requirements are achievable using split injections at ratios of 10:1, 50:1, or even higher. Increased flow through the inlet minimizes compound degradation (e.g. Endrin and DDT) and poor response for involatile compounds (e.g. PAHs, PCBs, dioxins and furans). In addition to fg-detection ECDs, sensitive GCxGC-TOFMS and GC-MS/MS are prime instrumental candidates for Shoot-and-Dilute GC.

Shoot-and-Dilute GC was tested by injections of a Used Motor Oil Composite Standard hundreds of times, interspersed with a polycyclic aromatic hydrocarbons standard (PAH) without changing the inlet liner. There is a slight baseline rise at the end of the run occurring over time likely from motor oil "heavies" eluting over several runs, but the responses for PAHs, even late eluting compounds, remained very stable.

P-61 A Simple, Rapid Method for the Analysis of Ethephon from Ketchup using Solid Phase Extraction and LC/MS/MS

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Ethephon is a small organophophosphate chemical that is metabolized by plants into ethylene, a potent plant growth regulator. Commercially, ethephon has been used on a broad range of fruits and vegetables, including cotton, tobacco, bananas, tomatoes, coffee, pineapples and berries, to accelerate the ripening process. Although the toxicity of ethephon to humans is relatively low, because it is used on such a wide range of foods intended for direct human consumption (e.g. ketchup), there is an increasing demand for monitoring ethephon levels in both the environment and in foods. In this study, we present a fast, simple, and sensitive method (low ppb) for the analysis of ethephon residues from tomato ketchup. Ethephon is first extracted from the ketchup matrix with a simple liquid extraction using acetone. This extract is then cleaned up using ion-exchange solid phase extraction (SPE), followed by a unique high-pH LC/MS/MS analytical method on an amino-type stationary phase. The amino-type stationary phase promotes retention of the extremely polar ethephon molecule (LogP = -0.57), allowing it to be separated from potential matrix interferences, while still maintaining a rapid (<5 minute) analysis time.

P-62 Using Large Volume Injection (LVI) on Conventional Split / Splitless Inlets to Improve Sensitivity or Reduce Sample Preparation

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Many labs are often asked to do more with less, whether this is to reach lower detection limits with current instrumentation or to do more work using fewer resources. Implementing a large volume injection (LVI) technique is one way to achieve either or both of these goals. LVI can improve detection limits by injecting more analyte on column. Alternatively, if less sample is processed, a larger injection can still result in the same amount of sample on column. LVI is typically performed using a specialized inlet though and has required an initial investment and / or instrumentation upgrades. This has made LVI out of reach to most labs. Significantly larger injections can still be performed using traditional split/splitless inlets though if using the correct parameters and hardware.

This work explores the requirements and limitations for LVI using traditional split/splitless injections. In some instances, injections up to 25 µL can be performed using existing hardware. This can significantly improve detection limits or dramatically reduce the volumes required for sample preparation. The effects of liner selection, solvent selection, column configuration, and oven temperatures are shown using commonly run EPA methods like PAH's and other semi-volatiles.

P-63 Rapid Analysis of Explosives Contamination in Soil Samples using Portable Micro-Thin Layer Chromatography

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For well over 100 years large scale manufacturing of explosive materials, global munitions test ranges, two world wars and many regional military conflicts, have created serious health and environmental issues through soil contamination. Common components of these explosives include ntiramines (HMX, RDX), nitroaromatics (2,4-DNT, TNT, TNB, Tetryl, Nitrobenzene), and nitrate esters (PETN, NG), along with environmental transformation products. Government organizations are responsible for site remediation including demining operations with requirements for safe, easy to use and reliable field-portable detection techniques for instant screening and characterization of soil. It is essential to distinguish among different compounds because the levels of clean-up for

different explosives can be set at different concentrations. One specific requirement is to rapidly distinguish between explosive aliphatic materials such as PETN and RDX for different soils. A range of detection and analysis results using a small, novel thin layer chromatography (TLC) unit will be presented. The microTLC platform can be deployed for the rapid separation and identification of low levels of explosive materials in soil samples, with typical limits of detection approximately 100 ng. Total analysis times are typically less than 5 minutes. The kit can be utilized for various explosive materials, including home-made explosive materials, military explosives, and propellant stabilizers. In addition, solid oxidizers with chlorates, perchlorates, bromates, nitrates, and nitrites can also be detected.

P-64 Neem: An Organic Pesticide

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Neem (Azadirachta indica) is an ever green tree indigenous to India. For centuries almost every part of this tree has been utilized across the agricultural, medicinal and personal care industries. The whole plant yields several thousand chemical constituents including, terpenoids, limonoids, fatty acids and essential amino acids. Three hundred natural products have been isolated so far, 135 biologically active from the oil alone. With the high number of active ingredients pest resistance is unlikely. Developing a method to analyze pure organic neem oil proved to be very difficult. Several different HPLC methods were used until the one with optimal separation was found. Reference standards were obtained for Azadirachtin A and B, Salannin, Nimbin, and 3-Desacetyl Salannin. Both the residue and the filtrate were shown to contain Salannin, Nimbin, and 3-Desacetyl Salannin.

P-65 Analysis of 200+ Pesticides in a Short LC Run Using Non-Timed SRMs on Triple Quadrupole Mass Spectrometer

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Increasing food safety concerns and the growing agricultural trade have resulted in stringent pesticide regulations globally. To comply with strict food safety standards, fast screening and quantitative methods for large numbers of pesticides are becoming important. Tandem quadrupole mass spectrometry offers highly specific and selective detection. However, it is also limited by intra-scan delays and dwell times required to get the maximum sensitivity and reproducibility. Therefore timed-SRMs are needed for analyzing large numbers of analytes. This poster describes a method for rapid analysis of 200+ pesticides/500SRMs in peach matrix using non timed-SRMs on a triple quadrupole mass spectrometer. A Thermo Scientific Hypersil GOLDTM column (100 x 3 mm, 3μm) was utilized for the separation of all analytes using a short LC run (10 minutes). The mobile phases were 0.1% formic acid in water and 0.1% formic acid in acetonitrile, respectively. Detection was performed using a Thermo Scientific TSQ Endura™ triple stage quadrupole mass spectrometer. The peach matrix was extracted with organic solvent using a QuEChERS method. One or two ion ratios were used to confirm each analyte. Calibration curves from 0.1 to 100 ppb were generated for the pesticides analyzed. This instrument is able to perform at 500 SRM/sec. data acquisition rate, which allows us to eliminate the need to set up a specific time window for each compound. 200+ pesticides were monitored throughout the entire LC run. The method development was significantly simplified. The experiment results will be discussed in detail.

P-66 Analysis of multiclass veterinary drug residues in pork meat and urine by ultra fast chromotagraphy with high performance triple quadropole mass spectormetry.

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The quantification of 50 multi-class veterinary drug residues from meat products and waste usually involves different extraction methods either with SPE or LLE extraction, which requires substantial time in both sample preparation and analytical run time. A new method, utilizing ultra fast chromatography and a triple quadrupole mass spectrometer will take advantage of a short run time on the LC/MS and to have a robust method to meet future regulation requirements and the posibility to do targeted screening. 10 uL injections of extracted meat and urine containing many veterinary drugs were injected onto C18 reverse phase column. Compounds of interest were eluted using a standardized fast gradient elution profile. A high performance triple quadrupole mass spectrometer with a heated electrospray source (HESI) was used to analyze the compounds of interest and the data are collected, analyzed, and reported using a customized software. A standard curve with seven points were prepared 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 above.

P-67 Increasing Extraction Efficiency of Pesticides & Dioxins from Wet Samples using a Novel New Polymer during Accelerated Solvent Extraction

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Accelerated solvent extraction (ASE) is a high-temperature and high-pressure extraction technique that is widely used for sample extractions in the environmental, chemical and food analysis industries. Extraction efficiencies when extracting volatile or semi volatile analytes from wet solids are often low, as the analyte of interest may partition between the extracting solvent and the water phase. Traditional pre or post extraction methods of heat evaporation cannot be used for volatile and semi-volatile compounds. Drying techniques that involve mixing the wet samples with an inorganic salt that has a high affinity for the aqueous phase are unsuitable for in-cell extractions. Drying methods with inorganic salts suffer from the limitations of clumping or precipitation making post extraction clean-up difficult. Off-line drying methods like freeze drying are extremely tedious and time consuming.

This study presents the use of a novel new polymer designed to remove moisture from wet samples like soil, tissue and food products and increase the extraction efficiency of volatile and semi volatile compounds. The polymer has a high capacity for water removal and does not suffer from some of the limitations of clumping or precipitation observed in some of the traditional drying methods. Data showing recoveries of organochlorine pesticides, polychlorinated dibenzo-p-dioxins, polychlorinated dibenzofurans, and polychlorinated biphenyls in different matrices will be presented.

P-68 Automated Solid Phase Extraction of Organochlorine Pesticides from Drinking Water

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Organochlorine pesticides are chlorinated hydrocarbon compounds that have a history of wide spread use both in the United States and globally. Organochlorine compounds degrade slowly and can bio-accumulate over time, with increasing concentrations in animals high in the food chain. Their ability to volatilize in warm regions allows them to spread over long distances, with measurable concentrations detected near the Arctic Circle and in alpine areas where they have not been used. Organochlorine pesticides have found their way into sediments and drinking water supplies posing serious health risks. Organochlorines have a wide range of both acute and chronic health effects, including cancer, neurological damage, and birth defects. In response to growing health concerns, the United States and Europe has banned several of these compounds such as DDT, dieldrin and chlordane. Others still in use include lindane, endosulfan and methoxychlor.

This study evaluated extraction recoveries of twenty five chlorinated pesticides from drinking water using automated solid phase extraction. This study also compared recoveries of the twenty five pesticides when using automated SPE to the traditional liquid-liquid phase extraction. The quantitative determinations of the twenty five chlorinated pesticides were performed by gas chromatography followed by electron capture detection (GC-ECD).

P-69 Direct Acetonitrile Injection for GC-MS/MS Analysis of Pesticide Residues in Tea

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QuEChERS (Quick, Easy, Cheap, Effective, Rugged, Safe) is a well known approach used for the extraction and clean-up of pesticide residues in various matrices. This procedure involved an initial step when a few grams of the sample are extracted with acetonitrile followed by a clean-up step (with dispersive-SPE) used to remove, to a certain extent, unwanted matrix compound (such as pigments, sugars, organic acids). Typically, the final extract ends up with the pesticides in acetonitrile. Acetonitrile is a polar solvent and can be problematic in GC-MS. Poor focusing of chromatographic peaks and high expansion coefficient are issues that need to be addressed when acetonitrile is used as a solvent for GC-MS analysis. To overcome this, an additional step can be added to the QuEChERS method where acetonitrile is replaced with solvents that are more amenable to splitless injections in GC-MS.

This work shows the results of the analysis of 21 pesticides in green tea using acetonitrile as final solvent and splitless GC injection. The compounds analysed are representatives of various classes of pesticides, such as carboxamids, OC, OP, pyrethriods, aromatic, phenylamides. The aim of this study was to assess the chromatography, repeatability, robustness and linearity of these compounds when using acetonitrile and splitless injections. For this, organically grown green tea samples were extracted with QuEChERS acetonitrile and spiked with the pesticides of interest at 10 ppb and 20 ppb. For all experiments a 1.0 µL sample volume was injected splitless into a Thermo Scientific TRACE™ 1310 GC coupled with a TSQ 8000 triple quadrupole GC-MS/MS analyser. Excellent chromatography for most compounds analysed was observed. The repeatability results at 10 ppb level show %CV below 9% (n=20). In addition to this, excellent linearity across 1-100 ppb matrix-matched calibration curve was obtained for all pesticides with minimum coefficient of determination (R^2) values of 0.996.

P-70 Laboratory Information Management System (LIMS) to improve quality control and quality assurance for PFOA and BPA methods

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Developing test methods that can analyze emerging contaminants and being able to detect these substances in a multitude of matrices is crucial. An innovative feature has been designed in MPI Research's proprietary LIMS, ExyLIMS™ that increases quality for this type of testing. This validated, Part-11 compliant feature, known as "batch module" groups analyte specific samples into batches for concurrent processing. Quality control (QC) samples are added to the batch automatically, background data is entered, preparation and analysis fortifications and standards are electronically documented while being physically prepared, and preparation and analysis results are recorded automatically from the instruments responsible for the analysis. Once the analysis results have been populated and calculations are final, the results are compared to the specification criteria of the analyte in question. Three levels of review and approval (analyst, peer verifier, and quality assurance auditor) are required in order for the batch to be completed and the report to be generated. This process has eliminated all transcription of sample weights, chromatographic data, sample identification and observations, and results. Validated electronic workbooks are created for each method, and this process has led to higher throughput, streamlined data processing by decreasing review times and final report output. This fully automated system allows for routine sample analysis on a daily basis, reduces data from many different platforms, and has maximized the number of tests for multiple analytes that are performed daily.

P-71 Monitoring of B-Lactam and Cephalosporin Antibiotics Residues Removal Using Chlorine Dioxide and Identification of the Breakdown Products by ESI and APPI LC/MS/MS

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B-lactams and cephalosporins are extensively used antibiotics, manufactured throughout the world. Many facilities manufacture multiple drugs, therefore, contamination at the manufacturing site presents a significant health hazard for anyone with a severe allergy to these antibiotics. Typical decontamination procedures use sodium hypochlorite solution, but this is not always completely successful and residues may be found on machinery and can be harmful to the machinery. To better decontaminate the equipment, a chlorine dioxide gas procedure was evaluated.

Chlorine dioxide conditions were optimized, based upon the disappearance of the parent antibiotic molecular ion. Swabbing procedures were developed and validated for the analysis of antibiotic residues from numerous surfaces. The removal of the antibiotic residue and identification of the degradation products was accomplished with accurate mass measurements and MS/MS from a q-TOF and with MS^n on an ion trap. Both electrospray positive and negative ion detection were used to look at the degradation products; carboxylic acid and amine containing products have the potential to form. In addition, APPI was used to detect the less polar degradation products. For each antibiotic studied, 3 to 7 degradation products formed. From the ten B-lactam and cephalosporin antibiotics evaluated, results showed that chlorine dioxide reduced the residue level by more than 3 orders in magnitude. The disappearance of the parent antibiotic residue does not verify that all the allergenic properties of theses antibiotics have been destroyed; therefore, this work focused on characterizing the products formed after chlorine dioxide degradation.

P-72 Qualitative and Quantitative Detection of Wheat, Barley and Rye Gluten in Beer by LC-MS

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Adverse physiological responses to gluten proteins from wheat, barley and rye affect millions of people worldwide. These proteins are implicated in a variety of food allergies, intolerances and immune responses, such as Celiac disease. People who suffer from these conditions must follow a strict dietary regimen, free of any gluten.

Beer is made predominantly from barley, but other gluten-containing grains are often used in the brewing process. Gluten-free beer production usually occurs in facilities where gluten-containing beer is also made. Contamination with gluten-containing grains can easily occur during the many processing stages from harvest to bottling.
Using mass spectrometry, we have identified physiologically relevant gluten peptides that can be used as markers for the presence

of trace levels of barley, wheat and rye in beer. A proteomic approach was used, whereby proteins extracted from beer samples at various stages of production were enzymatically digested with pepsin, trypsin and chymotrypsin. LC-QTOF accurate mass and MS/MS analysis permitted the identification of several marker peptides representing both intact and hydrolzed gluten from each grain and allowed accurate and sensitive detection of each marker into the ppb range. A key aspect of this work is a new calibration method that relates marker peptide concentration to gluten concentration, as required by the new FDA guidelines. Isotopically labeled synthetic peptides were used as internal standards.

P-81 Novel HPLC Method Integrates Analysis with Automated Sample Clean-up for Aflatoxins and Ochratoxin A

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The analysis of food and feed consists of sampling, extraction, clean-up and analysis. For clean-up, the use of immunoaffinity columns is very common because of the specificity for mycotoxins, but can be time consuming -- a typical IAC column can tolerate a flow rate of about 2 ml/min and it could take upwards of 30 minutes to extract and clean up a single sample. Mycotoxins are usually eluted from the IAC by organic solvents, which denature the antibody and release the toxins. For analysis by HPLC small injection volumes must be used to avoid poor chromatography on the reverse-phase column. A new technique was developed which uses thermal denaturation and water to release mycotoxin from the IAC. New column types for rapid clean-up based on immunoaffinity columns were developed for Aflatoxins and for Ochratoxin A, which could be combined with the thermal denaturation. Not only is this faster, but water is used as the eluant from the immunoaffinity column. The switch from solvent to water allows for large-volume injection directly onto the HPLC column. The online clean-up and analysis is automated by use of the ThermELUTE module on the FREESTYLE automated sample prep system. In combination with online injection, analytical sensitivity can be increased without manual manipulation. The increase in sensitivity provides a reduction of sample load, while maintaining excellent chromatographic results, which in turn allows for analysis of mycotoxin levels well below regulatory limits.

P-82 Multiclass multiresidue Analysis of >100 Veterinary Drug Residues in Bovine Tissues by Filter-Vial Dispersive-SPE and LC-MS/MS

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High-throughput analysis is needed to meet demand for monitoring of chemical contaminants in foods, such as veterinary drug residues in food animal tissues. The current veterinary drug residue monitoring method used by the USDA Food Safety and Inspection Service (FSIS) uses a combination of hexane-partitioning, dispersive-SPE, and solvent evaporation to achieve adequate cleanup for 20 mg equivalent sample injections to meet regulatory detection limit needs. This extra effort adds to the time and cost of the method and limits sample throughput. In this study, a new LC-MS/MS Q-Trap instrument was used which met the same detection limit needs with merely 0.17 mg equivalent sample injection. This obviated the hexane-partitioning and solvent evaporation steps, and filter-vial dispersive-SPE was used to eliminate a centrifugation step. In this way, sample turnaround time was 20 min for an individual sample, and sample throughput was doubled for a batch of 60 samples. More than 100 of the 130 tested drug analytes achieved 70-120% recoveries and <20% RSD for 90 replicate injections at 3 spiking levels over the course of 3 days in each of bovine kidney and muscle sample types. This method outperforms the current FSIS method and may be used in the future to provide quantitative as well as qualitative screening results in routine monitoring programs.

P-83 Sensitive and Fast Analysis of Aflatoxin M1 in Milk at Picogram Levels using Interference Removal Solid Phase Extraction and LC-MS-MS Analysis

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Aflatoxins are considered potent carcinogens and are regulated in food and animal feed. If digested by milk-producing animals, Aflatoxin B1 is metabolized to form Aflatoxin M1, which in turn can then be released into milk. Because of its carcinogenic effects, low regulatory levels are set for Aflatoxin M1 in milk (25-50 pg/mL). Sensitive methods are required for such testing. In addition, the methods should be able to remove matrix interferences for successful detection and analysis.
The presented method demonstrates the analysis of Aflatoxin M1 in milk at 25 pg/mL. Milk is initially centrifuged to remove fats and solids. The sample preparation strategy includes extraction of M1 from milk using the salting out effect, cleanup of the extracted sample by simple pass through an SPE cartridge and subsequent analysis by LC-MS-MS. The interference removal strategy is easy and fast. The method demonstrates acceptable recoveries above 75% at 25 pg/mL and 250 pg/mL spiking levels.

P-84 Analysis of Patulin in Apple-Based Products using Molecularly Imprinted Polymer Solid Phase Extraction and Fast UHPLC Detection Method

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Patulin is a mycotoxin produced by a number of fungal species in rotten fruits, specifically, apples. A sensitive and fast method for low level patulin detection in apple juice and apple puree is demonstrated herein. The sample preparation methodology utilized molecularly imprinted polymer (MIP) cleanup allowing for easy and effective separation of patulin from matrix interferences. MIP solid phase extraction material is engineered to selectively bind the analyte of interest while strong washes can be applied to remove other matrix components resulting in cleaner samples. Analysis of patulin was done by UV detection in less than 7 minutes using HPLC column packed with sub-2 micron monodisperse porous particles. The method was demonstrated for apple juice samples spiked at 50 ng/mL and 10 ng/mL and for apple puree samples spiked at 10 ng/mL.Excellent patulin recovery and reproducibility was observed from the various matrices and will be described.

P-85 Analysis of Sulfonamides, Trimethoprim, Fluoroquinolones, Quinolones, Triphenylmethane Dyes (and their Leuco Metabolites) and Methyltestosterone in Fish and Shrimp Using LC-MS/MS

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An LC-MS/MS screening method is described for the detection and identification of 26 drugs of interest in fish and other aquaculture species. The analytes include: 13 sulfonamides, trimethoprim, three fluoroquinolones, three quinolones, three triphenylmethane dyes, with two leuco dye metabolites, and methyltestosterone. Frozen ground tissue is mixed with pH 4.5 EDTA-McIlvaine buffer and subsequently double-extracted with acetonitrile, *p*-toluenesulfonic acid and N,N,N',N'-tetramethyl-*p*-phenylenediamine dihydrochloride. The extracts are analyzed using the Agilent 6490 LC-MS/MS with dynamic multiple reaction monitoring. The procedure was validated as both a quantitative and semi-quantitative screening method for multiple fish and shrimp matrices. For screening, a single-point tilapia matrix-extracted standard was used as a comparison reference standard to which all extracts (of any matrix) were compared. The screen was applied to eight types of fish (catfish, eel, pangasius, sablefish, tilapia, swai, salmon, and trout) and shrimp at the level of concern.

P-86 Determination and Confirmation of the Antiviral Drug Amantadine and its Analogs in Chicken Jerky Pet Treats

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A method for analyzing the antiviral drug amantadine and its analogs, rimantadine and memantine, in chicken-based pet treats is described. Chicken treats were extracted with water and acidic acetonitrile followed by dispersive sorbent cleanup. Two complementary liquid chromatography - mass spectrometry (LC-MS) procedures, ion trap and high resolution (HR) MS, were used to detect and identify residues. Matrix blanks and samples fortified at 2.5 - 50 ng/g were analyzed. Qualitative validation for all compounds at all fortification levels was successful. With the ion trap LC-MS^n method, the abundance ratio of ions in the MS^3 product ion scan was used to establish confirmation of identity. The presence of two ions specific for each compound in the HRMS analysis also confirmed the identity of these residues. The quantitative validation focused on amantadine, and a deuterated internal standard was added to samples prior to extraction. Average recoveries for amantadine in fortified samples (n=64) using the ion trap LC-MS method ranged from 76 - 123% with relative standard deviations ≤ 12%. Quantitative results from the HRMS method generally matched the ion trap LC-MS data. The procedure was not optimized for the quantification of rimantadine and memantine, but was able to detect the presence of these compounds at levels greater than 2.5 ng/g. The method was also tested on several chicken pet treat test samples. Amantadine was detected and identified in these samples at levels ranging from < 2.5 ng/g to over 600 ng/g. Rimantadine and memantine were not detected in any samples.

P-87 Liquid Chromatography/Fluorescence Detection of Avermectins in Bovine Milk

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A rapid method for the simultaneous isolation and subsequent detection via liquid chromatography/fluorescence detection (LC/FLD) of Eprinomectin **(**EPR), Moxidectin (MOX), Doramenctin (DOR), Emamectin (EMA) and Ivermectin (IVR) in bovine milk is presented. Isolation of the five compounds is achieved through acetonitrile extraction followed

by dispersive solid phase extraction (dSPE) clean-up with primary-secondary amine (PSA). The extract is derivatized with trifluoroacetic andhydride (TFAA) making the sample ameable for LC/FLD. Data was also obtained utilizing PSA column SPE clean-up instead of dSPE. The optimization of the LC/FLD conditions results in chromatographic runs of less than ten minutes. Recoveries from milk samples fortified at 1, 5, and 10 ng/mL (ppb) ranged from 70 to 110%. The confirmation of residues in samples can be accomplished by re-extraction of a suspect sample via this method without derivatization and analyzing the extracts by LC/MS. For the purposes of this publication, fortified sample extracts at each fortification level were extracted (without derivatization) by this method and injected on a LC/ion trap mass spectrometer. Results from these injections were equivalent to those obtained by HPLC/FLD. This method provides a rapid, specific, robust, and simple analysis for the presence of avermectins in bovine milk matrices with minimal solvent usage.

P-88 A Quick Assay for the Quantitation of Deoxynivalenol in Grain Samples by Liquid Chromatography with UV Detection

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A quick and effective extraction of Deoxynivalenol (DON) in food samples is described. The samples were extracted with an 80% acetonitrile/water solution, followed by a second extraction using an 80% methanol/water solution. The extract was then subjected to an immunoaffinity column (IAC) for clean-up. Once the analyte was isolated, quantitation was then obtained by Liquid Chromatography (LC) with UV detection at 220nm. LC/UV parameters were optimized with a Phenomenex Kinetex C18 LC column resulting in a 6-minute run time.

Certified reference materials (CRMs) of three different matrices, (barley, corn, and wheat) at three different levels were analyzed. For the lowest level, a wheat CRM sample containing 0.5 parts per million (ppm) was analyzed. The other CRM samples included barley at 1.0 ppm and corn at 1.9 ppm. All recoveries were calculated using an external standard curve. Recoveries from all CRM samples ranged from 80% to 102%. Confirmation for DON in the sample extracts at each level was accomplished by injecting the samples in an ion trap mass spectrometer.

This method provides a rapid, specific, robust, and easily controlled assay for the analysis of DON in food samples with minimal solvent usage. This extraction reduces the consumption of organic solvents by at least 60 % when compared to methods currently used by FDA laboratories. Other existing methods that do not use immunoaffinity clean-up columns produce dirty extracts that require chromatographic column clean up steps after each sample injection. The method presented here did not require such clean up steps, which lead to faster analysis time per sample, which minimizes the solvent consumption, and waste production per injection.

P-89 Dopant-Assisted Atmospheric Pressure Photoionization of Patulin in Apple Juice and Apple-Based Food with Liquid Chromatography-Tandem Mass Spectrometry

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A dopant assisted atmospheric pressure photoionization (APPI) with liquid chromatography tandem mass spectrometry (LC-MS/MS) method was developed to determine patulin in apple juice and apple-based food. Different dopants, dopant flow rates, and LC separation conditions were evaluated. Using toluene as the dopant, the LC-APPI-MS/MS method achieved a linear calibration from 12.5 to 2000 µg/L (r^2>0.99). Matrix-dependent limits of quantitation (LOQs) were from 8 µg/L (solvent) to 12 µg/L (apple juice). [^{13}C]-patulin fortified apple juice samples were directly analyzed by the LC-APPI-MS/MS method. Other apple based food was fortified with [^{13}C]-patulin, diluted using water (1% formic acid), centrifuged and filtered, followed by LC-APPI-MS/MS analysis. In clear apple juice, unfiltered apple cider, applesauce, and apple-based baby food, average recoveries were 101±6% (50 µg/kg), 103±5% (250 µg/kg), and 102±5% (1000 µg/kg) (ave±SD, *n=16*). Using the suggested method, patulin was detected in 3 out of 30 collected market samples with concentrations ranging from <LOQ to 18 µg/L. The use of [^{13}C]-patulin allowed quantitation using solvent calibration standards with satisfactory precision and accuracy.

P-90 Analysis of Pesticides in Olive Oil Using a Modified QuEChERS Method with LC-MS/MS and GC-MS/MS

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A simple and high-throughput screening method for the analysis of pesticides in olive oil by LC-MS/MS and GC-MS/MS is presented. A modified QuEChERS sample preparation method was developed to improve the extraction recovery of highly lipophilic pesticides.

The acetonitrile (MeCN) extract of the olive oil was directly injected to LC-MS/MS, while other GC-amenable compounds were treated with the modified QuEChERS procedure for GC-MS/MS analysis. The average recoveries for 80 pesticides quantified by LC-MS/MS at 200, 500, and 1000 ng/g fortifying levels were 91% or better (RSD < 5.5%), while GC-MS/MS analysis demonstrated 81 % or better (RSD < 7.2%) for average recovery from 59 compounds at the same spike levels. This method demonstrated the improved recovery of the several challenging lipophilic pesticides in olive oils.

P-91 Survey of Sudan Dyes in Palm Oil and Method Validation by LC-MS/MS and LC-UV

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Sudan I, II, III, and IV dyes are banned for use as food colorants because they are toxic and carcinogenic. These dyes have been illegally used as food additives in products such as chili powder, worchester sauce, and palm oil to enhance their red color. Since 2003, the UK's food safety agency (FSA) has recalled over 350 products that have been adulterated with Sudan dyes. An imported palm oil sample purchased in the US was tested and found to contain more than 50 ppm of Sudan IV. In order for the agency to further investigate palm oil adulteration with unapproved color additives, analytical methods were validated to identify and quantify Sudan I, II, III, and IV. These methods consist of an LC-UV method for ease of analysis and an LC/MS/MS method for confirmation. Sample clean-up using solid phase extraction was optimized using Alumina B cartridges and method recoveries ranged from 92-112%. Method validation was performed using three separate palm oil samples at three concentration levels. According to the FDA Foods Program Guidelines for the Validation of Chemical Methods recovery results are required to fall between 70-125% and replicate samples should have relative standard deviations (RSDs) less than 15%. The method limit of quantification (LOQ) in palm oil was 2 µg/kg using an AB SCIEX Triple Quad 4500 LC-MS/MS. Additionally a survey of palm oil products in the Washington DC area was performed to determine if there are other adulterated samples on the market.

P-92 Comparison of Three Analytical Methods for Sulfite Determination in Challenging Food Matrices

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Sulfites are a family of preservatives added to foods in order to limit browning and microbial growth. Sensitive individuals have reported severe allergic reactions as a result of sulfite consumption. In 1986, the US FDA mandated that sulfites be declared on the label of any product containing in excess of 10 ppm sulfite. Furthermore, the FDA stated that the AOAC Official Method #990.28, the optimized Monier-Williams (MW) method, be used for the quantitation of sulfites in foods. For most sulfite-containing matrices, the MW method gives accurate results. However, vegetables from the *Allium* (garlic) and *Brassica* (cabbage) genera produce false positive results due to the presence of high levels of endogenous sulfur compounds. A modified MW method has been published which included changes to assay conditions to ensure more specificity and selectivity. This method has demonstrated success with garlic samples. Recently, an LC-MS/MS method was developed and validated for determining the sulfite content of food matrices by converting sulfite into a stable formaldehyde adduct, hydroxymethylsulfonate (HMS). Further investigation was needed to determine if false positives existed with this new method. Food matrices known to cause false positives with the MW method (garlic, wasabi powder, onions, shitake mushrooms and broccoli) were investigated by all three methods. The results are compared to determine the extent of false positive responses for each method. The ability to reduce false positives will enable accurate determination of added sulfite to ensure compliance with sulfite labeling requirements. This will help sensitive individuals avoid inadvertently ingesting sulfites.

P-93 Factors Affecting the LC-MS/MS Analysis of Pesticide Residues in Crops

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The need for selective and sensitive analyses of pesticides in crops and food has led to the development of QuEChERS methods combined with LC-MS/MS analyses. This presentation will highlight results from an ongoing method development project in which a general QUEChERS extraction followed by LC-MS/MS analysis was developed to investigate a suite of structurally and functionally variable pesticide residues in different types of crops. Target compounds included an organophosphate pesticide (diazinon), a neonicotinoid insecticide (acetamiprid), a carbamate pesticide (carbaryl), an amide herbicide (propyzamide) and a triazine herbicide (atrazine). Crop matrices included brown rice (high carbohydrate), kale (high pigment), egg (high protein), lime (high acid), corn (staple crop), and celery (high moisture). The matrix of pesticide and crop combinations provided the ability to assess both the extraction and analysis for specificity and sensitivity. The aim of the study was 1) to meet typical method guidelines; and 2) to investigate factors affecting the choices that are made during method development (e.g. solvent calibration versus matrix-matched calibration, the use of internal standard, etc.). In general, both solvent and matrix-matched calibrations were linear with r^2 values

>0.99, though significant ionization suppression in the corn and lime matrices across all target compounds necessitated the use of matrix-matched calibrations for determining concentration. Additionally, recoveries of target analytes were 80-110% in the celery, corn and kale samples, but ≤50% in the egg, lime and rice samples. Other details of the method aimed at understanding the factors that affect the method will be presented in the poster.

P-94 Multi-component quantitative analysis of pharmaceuticals in the environment by UHPLC-MS/MS with on-line SPE

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Pharmaceuticals comprise a group of emerging contaminants which have received considerable attention in recent years.

Many common drugs can be found in the environment and sometimes even in drinking water. These drugs and their metabolites get into the waste water through excretion via the urine or feces and may reach surface water, groundwater and also drinking water after the passage in the sewage treatment plants. So far, conventional sewage treatment plants are failing to eliminate the Biodegradable substances completely.

Many of these compounds are ubiquitous, persistent and biologically active with recognized endocrine-disruption functions. Paying attention to the hazardous nature of these compounds, there is a need to provide fast and sensitive multi-residue methods that are able to analysis multiple classes of compounds within one analytical procedure. Highly sensitive triple-quad-MS systems are suitable tools for the analysis of residues in ground-, surface- and wastewater, but development of a simple, fast and reliable method for simultaneous measurement of trace levels of various different classes of analytes in complex matrices is a challenge.

This study describes a novel multi-residue UHPLC-MS/MS method that utilizes an online SPE enrichment of the various compounds followed by a fast and optimized chromatographic gradient which results in excellent ng/L detection levels.

With online SPE no further sample pre-treatment is necessary but the transition of the low pressure online SPE part of the analysis to the high pressure analytical part is difficult. Using the benefit of the modular design of Shimadzu's Nexera X2 combined with the high speed values for MRM recording and the fastest polarity switching time of 5 ms on the Shimadzu LCMS-8050, the difficulties of analyzing various classes of compounds in different polarities during one single analysis in sufficient sensitivity could be overcome.

P-95 Analysis of Pesticides in Baby Food Using a Triple-Quadrupole GC/MS/MS, Part II

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Contamination of food products with pesticides is a growing concern because of recognized adverse health effects, increasing world-wide usage of pesticides, and increasing imports of raw foodstuffs from foreign sources. Gas chromatography mass spectrometry (GC/MS) has been used extensively to quantify trace level pesticides in food matrices; the most significant challenges have been matrix interference and achievement of meaningful health-based detection limits for the compounds of interest. The QuEChERS (Quick Easy Cheap Effective Rugged and Safe) sample preparation method has helped to overcome some of the problems of matrix interference, and commercialization of QuEChERS kits has promoted widespread screening of foodstuffs for trace pesticides. Triple quadrupole GC/MS/MS operating in the Multiple Reaction Monitoring (MRM) mode has emerged as the technique of choice for analysis of trace level contaminants in complex matrices.

This poster is the second in a series which presents instrument configuration, operating parameters, and analytical results for analysis of trace levels of 37 pesticides of various chemical classes in a QuEChERS extract of baby food using the MRM mode on the Shimadzu triple quadrupole GC/MS/MS. Results were evaluated for calibration linearity, analytical precision, and accuracy. The effect of MS resolution on compound selectivity in a complex food matrix is also discussed.

P-96 Screen for Over 470 Residual Pesticides the Same Day your Triple Quadrupole GC-MS/MS is Installed!

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To insure food safety, the number of pesticides monitored in agricultural products has increased to several hundred. To determine the pesticide concentrations, calibration standards are prepared over a range of concentrations and analyzed using GC-MS or GC-MS/MS, and calibration curves are generated from the data. Method development and calibration can be tedious and time-consuming, especially when analyzing several hundred pesticides. Moreover, some pesticide standards cannot be easily obtained because of import or safety restrictions.

To address these problems, Shimadzu has developed the Pesticide Quick DB, a software product which provides semi-quantitative screening and reporting for over 470 pesticides without the need for pesticide standards, method development, or instrument calibration. The Pesticide Quick DB supports highly accurate, semi-quantitative determination of pesticide concentrations by using 19 isotopically labeled pesticides as internal standards, one designated for each specific compound class, and second-column confirmation without venting the MS. Acquisition methods for GC-MS by Scan/SIM and for GC-MS/MS by Scan/MRM, provide quantitation by SIM or MRM, respectively, with simultaneous confirmation by matching the acquired scan spectrum to standard library spectra. The Pesticide Quick DB includes retention indices for two different capillary columns, standard mass spectra, and calibration curves for over 470 pesticides.

This poster will demonstrate that the Pesticide Quick DB allows rapid, semi-quantitative results when screening for over 470 pesticides, without the need for hundreds of pesticide standards. The Twin Line MS Kit and simultaneous Scan/MRM measurements added the ability to confirm the identity of the targeted pesticides as well as the calculated concentrations.

P-97 Fast GC-MS/MS Analysis Of Multicomponent Pesticide Residues (360) In Food Matrix

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The potential of this approach is demonstrated in the actual study by analyzing 360 pesticides in apple QuEChERS extract in less than 10 minutes. Contamination of food products with pesticides is a growing concern because of recognized adverse health effects, increasing world-wide usage of pesticides, and increasing imports of raw foodstuffs from foreign sources. Consequently, the number of samples as well as monitored pesticides became significantly higher in the last decade. To handle this high sample load, a Quick, Easy and Cheap cleanup procedure called QuEChERS was established. Unfortunately, samples prepared by this method contain large matrix signals which complicate accurate pesticide quantification. Due to this drawback the use of tandem MS instruments using multiple reaction mechanisms (MRM) became more frequent in the last years, as it increases selectivity and sensitivity. Beside matrix interference the analysis time is a crucial point when handling a high sample load in routine work. Combining the speed of fast GC and the selectivity of tandem MS is a powerful tool to increase laboratory efficiency and reduce working costs. As fast GC reduces the peak width at half height (FWHM) down to about 1 s the detector must be able to follow sharp increases of signals. Therefore, fast MRM switching modes with no interfering cross talks are needed.

P-98 Quantitative analysis of pesticides in QuEChERs extracts using APGC/MS/MS

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Pesticides are widely used in the production of fruit and vegetables across the globe. With over 1000 pesticides in use, laboratories are under pressure to increase the range of pesticides determined. Typically, this analysis is carried out using a dedicated GC-MS/MS system with an EI source. High energy imparted by the EI process can cause extensive fragmentation. APGC is a soft, chemical ionization technique which results in high relative and absolute abundance molecular ions and, therefore, increases both sensitivity and specificity. Strawberry, pear, tomato and spinach samples were extracted using a QuEChERs protocol. To test the repeatability at low concentration, each matrix was fortified with the pesticide mix at 1 µg/kg. Two MRM transitions were monitored for each pesticide. The analysis of 20 GC amenable pesticides, chosen due to the challenges they present to EI systems, was achieved using APGC. The enhanced signal of the molecular ion enabled sensitive and specific measurements. The response for each pesticide was linear from 0.05 to 50 ng/mL with correlation coefficients R^2 of >0.997. The limits of detection, ranging from 0.01 to 0.5 ng/mL are well below the regulatory requirements. To assess the accuracy and precision of the method, each sample matrix was spiked at 1 µg/kg (10x below MRL) and ten replicate injections made. All pesticides were within 5% of the true concentration. The %RSDs for all pesticides in all four matrices were < 5%. This demonstrates that the method is both accurate and reproducible across different sample matrices analyzed on different days.

P-99 Advances in Screening Capability for the Detection of Residues and Contaminants in Food Using Accessible Mass Detection

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Global food laws require that the composition of processed foodstuffs is declared on the packaging and that it is free from agricultural chemicals and contaminants below relevant regulatory limits. The diverse physiochemical properties of such chemicals have dictated the need for a variety of analytical techniques to effectively control the food chain. There is an increased need for

cost-effective, robust and broad-scope screening platforms, which can be implemented in routine control laboratories. Important considerations are the flexibility of scope and the extent of compliance with internationally recognized performance criteria. A mass detector, the size of a PDA detector has been recently developed as a result of patented technology; it's applicability as a screening tool compliant with regulatory requirements is demonstrated.

Extracts of processed foodstuffs were prepared using conventional procedures such as solid phase extraction, solvent extraction and immunoaffinity clean-up (as applicable for the commodity/ analyte combination) prior to analysis. The UPLC® Cortecs UPLC C18 (1.6 µm, 2.1x100 mm) was utilized. The extracts were analyzed using the ACQUITY UPLC® coupled to an ACQUITY QDa Detector, a single quadrupole mass detector with a pre-optimized electrospray ionization (ESI) source. The detector performance is shown to be sensitive and robust providing advantages over conventional screening methods routinely used within the food industry such as enhanced analytical performance, ease of use and flexibility allowing multiple methods to be consolidated within a single method.

P-100 Analysis of Glyphosate, Glufosinate and AMPA in Bottled, Tap and Surface Water Using Time De-Coupled Chromatography

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The analysis of Glyphosate and AMPA were conducted using an automated Time De-Coupled LC/LC/MS/MS system. Since glyphosate does not have any chromaphores, a derivatization step is usually required to ensure trace level detection. With electrospray detection, glyphosate usually shows a weak response. The analysis started by filtering 10 ml water samples in a 20 mL vial. The reaction was performed directly in the autosampler. The samples were derivatized with an FMOC procedure, by adding 5 mL of acetonitrile, 0.5 mL of a borate buffer (pH 9) and 0.5 mL FMOC-Cl reagent. The reaction was completed after 60 minutes and quench by adding 1 mL of 2% phosphoric acid. A 0.5 mL aliquot of glyphosate-FMOC was aspirated and enriched on a trap column. With a Time de-Coupled solution, the analysis of glyphosate and AMPA in water was performed using 3 automated and programmable sequences. The first part of the analysis performed the conversion of glyphosate and AMPA with the FMOC derivative. The second part of the analysis used an automated sequence for quenching the reaction with an automated addition of 1 mL of 2% phosphoric acid. The final part of the analysis used an AT-column dilution function for high volume injection of the water:acetonitrile (66:33) sample. Up to 5 mL of derivatized sample was loaded onto a trap column. The trapped analytes were analyzed on a high resolution column using a back flush gradient. With this automated solution, glyphosate, glufosinate and AMPA were detected at ppt level (100 ng/L).

P-101 Quantitative Analysis of Pesticide Residues in Rice using LC-MS/MS

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Rice is one of the most consumed food in the world and its demand has increased in recent decades. Basmati rice is a variety of long grain rice which is traditionally cultivated in northern part of India. To improve its production yield, the use of various pesticides in various stages of cultivation has increased. Due to the adverse effects of these pesticides on human health and to the environment, use of pesticides must be controlled and monitored. A multi-residue method has been developed for the analysis of most commonly used pesticides in Basmati rice. In this work, large numbers of pesticides were evaluated on LC with tandem quadrupole mass spectrometry. Two MRM transitions were monitored for quantification and identification of all the pesticides that were studied. A simple sample preparation along with rapid detection method was employed to analyze rice samples. The recoveries for all pesticides in rice were calculated at legislative limits. Varieties of basmati rice and other rice samples were studied and results will be reported.

P-102 A Rapid analysis of Sudan And Other Prohibited Dyes In Chilli Powder Using Ultra Performance Liquid Chromatography and Tandem Mass Spectrometry

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Sudan dyes are synthetic dyes frequently used for coloring plastics and other synthetic materials. However, due to their intense color, these dyes have been fraudulently used to enhance the color of various food products such as spices, mixtures of spices and sauces. According to the International Agency for Research on Cancer (IARC), the Sudan and azo dyes have both been classified as potential genotoxic and/or carcinogenic substances. Since the illegal use of these dyes constitutes a risk for public health, detection using simple, sensitive and reliable analytical methods is extremely important. A fast and sensitive method for the detection and quantification of Sudan and azo dyes will be presented. The separation and detection of 11 Sudan and azo dyes was performed on high resolution chromatography coupled with tandem MS technology. The separation was achieved on a reversed-phase column with a 12 minute gradient method. MRM data was collected for all dyes in ESI positive mode with two transitions for each analyte.

The samples analyzed in this work were chilli powders and curry powders that are known for their risk of containing Sudan dyes. The samples were extracted and injected for analysis. Linearity of the method was studied with solvent and matrix match spiked calibration curves. Recovery was calculated based on a 10 µg/kg (10 µg/L) spike in blank chilli powder. The data on recovery and matrix effects will be presented. Various chilli and curry powders were analyzed with this method and results will be reported.

P-103 Determination of Pesticide Residues in Oleoresins: Optimized Sample Preparation Prior to LC-MS/MS and GC-MS/MS Analysis

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In recent years, food safety laboratories have adopted new and simplified sample preparation methods designed to reduce analysis time and related costs, as well as to increase throughput. For example, the QuEChERS methods for fruits and vegetables require only minutes for sample preparation and replace prior methods that took hours or days. This type of simplified sample preparation, with modification, has also been applied to fatty matrices such as nuts and oils. In this presentation, we will discuss the determination of pesticides in spice oleoresins. Oleoresins contains the highly concentrated oily and resinous materials obtained from natural spices along with vegetable oils added to produce a consistent product. Because the oleoresins themselves are so highly concentrated, very high amounts of matrix co-extractives are present in solvent extracts of these materials. Therefore, more rigorous cleanup is required compared with standard QuEChERS procedures. Alternative extraction and cleanup procedures for oleoresins are under investigation and will be presented and discussed. Analysis is performed using tandem LC-MS and tandem GC-MS. A new GC-MS technique using atmospheric pressure mass-spectrometry (APGC) is employed for the tandem GC-MS analysis.

P-104 Determination of Sudan Dyes in Chili Oleoresin: Optimized Sample Preparation Prior to LC-MS/MS Analysis

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The Sudan dyes are a class of diazo-conjugate, water-insoluble compounds used to impart colors to waxes, oils, solvents and plastics. Many of these dyes individually or in mixtures produce colors very similar to colored compounds found naturally in red chili peppers. Unfortunately the Sudan dyes, potential carcinogens, are sometimes illegally used to enhance the color of chili based products. Chili oleoresin is a highly concentrated mixture of natural oils and resins extracted from chili. It is used as both an intense flavoring agent and an intense coloring agent in the food industry. It is also used as a source of capsaicin for medicinal preparations and in pepper spray. Since the oleoresin is used as a food coloring agent and for other human consumption uses, an effective analytical method is required for detection of illegal dyes in this product. Prior analytical methods focused on four phenolic Sudan dyes (Sudan I, II, III and IV). This poster presents an SPE based analytical method suitable for the LC-MS/MS determination of those phenolic Sudan dyes along with six other important dyes including polyphenolics (such as Sudan Orange G) and amines (such as Sudan Black).

P-105 Discovery of Pesticide Protomers Using Routine Ion Mobility Screening

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Criteria to instill confidence in pesticide identification include acceptable product ion ratio tolerances and relative intensities of the detected ions. Ion ratio performance can vary with instrumentation, matrix and sample concentration. SANCO/12571/2013 guidance document describes method validation and analytical quality control requirements to support the validity of data in compliance with maximum residue limits, enforcement actions, or assessment of consumer exposure to pesticides in the EU. Here we use ion mobility mass spectrometry to gain greater understanding of ion ratio variation.

Empirically isobaric pesticide protomers have been identified and characterised using ion mobility. It has been possible to separate protomers (ions different only by protonation site) and determine their respective collision cross section values and individual protomer fragmentation dissociation pathways. This enabled unique visibility of product ion formation information, enabling the product ions to be selected that will result in improved product ion ratio reproducibility. UPLC-HDMS^E experiments were performed on a Synapt G2-S using a series of standards, spiked matrices and previous proficiency test samples.

For indoxacarb, determined to be present in proficiency sample FV-13, two mobility separated species with CCS values of 136.49Å^2 and 147.94Å^2 were obtained, thought to be ions generated by multiple sites of protonation. Fragmentation spectra generated from the mobility separated protomers allowed identification of distinctive and common fragments. Another example of protomer formation was observed for fenpyroximate. Each protomer respectively produced one of the two most abundant fragment ions. Ion ratios obtained by conventional MRM analysis for this compound could be affected by protomer formation.

P-106 The Combining of an Integrated Microfluidic Device with Collision Cross Section (CCS) Ion Mobility Screening for the Analysis of Pesticide Residues in Food

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Full spectra acquisition and the specificity of accurate mass measurement is well characterised. It is used in combination with time tolerances, isotopic matching, fragment ions/ratios and response thresholds to help reduce false positive and false negative identifications in screening assays. Advances in mass spectrometry have vastly improved sensitivity for full spectral analysis, but further sensitivity enhancements would improve the mass spectral data quality. This is especially important to avoid compromised precursor ion or fragment ion information, and ensure high mass accuracy below the legislation levels. The integrated microfluidic device was interfaced to a Synapt G2-S mass spectrometer operating in ion mobility data acquisition mode, enabling enhanced sensitivity and selectivity to be obtained for the sample acquisitions.
Initially, ion mobility data was acquired using the integrated microfluidic device, for a series of solvent standard mixtures to generate retention time and collision cross section (CCS) information for pesticides. This subsequently enabled the correct identification of the pesticide residues in the matrix matched samples and proficiency samples. Compared to previously obtained results using conventional UPLC, gains in both sensitivity and signal to noise, with excellent linearity correlation coefficients, were obtained for the matrix matched calibrants ($r^2 \geq 0.95$). Improvements in sensitivity enabled matrix dilution to be performed and detection of 1pg on column to be obtained. CCS measurements obtained during the UPLC ion mobility acquisitions were used to rapidly determine the retention times of the pesticide solvent standards and identify the residues present in a previous proficiency sample.

P-107 Are Pork Residues Present in My Gummy Bears? Gelatin Speciation by LC-MS/MS

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Following the Food Standards Agency (FSA)'s announcement in January that horse and pig DNA had been identified in beef products sold by several supermarket chains, further testing across Europe and beyond has revealed widespread incidences of such food contamination. This intended adulteration for financial gain or careless false declaration of meat products is a severe problem for consumers who have ethical or religious concerns about the consumption of pork or horse, more specifically the Muslim or Jewish communities who represent about 23 % of the worldwide population. As the tolerance level for porcine and equine content in foods is 0 %, for religious reasons, the limit of detection (LOD) needs to be as low as possible and so the continued development of more sensitive methods is necessary.

However, pork based products are not only used as the meat but can also be found in gelling agents in food (for example in candy, ice cream, and marshmallows) as well as in the cosmetic and pharmaceutical industry in the form of gelatin. Gelatin is made from collagen, a protein, which has been extracted from the skin, bones, and connective tissues of animals such as cows, chicken, pigs, and fish.

Here we present the results from the initial development of an LC-MS/MS method utilizing AB SCIEX TripleTOF® 5600 and 4000 QTRAP® LC/MS/MS systems for the determination of the origin of gelatin used in food products and also pharmaceutical capsules.

P-108 Target and Non-Target Accurate Mass Screening for Pesticides using LC-MS/MS

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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 the AB SCIEX TripleTOF® system operated in high resolution accurate mass MS-IDA-MS/MS 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 MasterView™ software.

P-109 Identification, Quantitation and Confirmation of Pesticides in Food Samples using 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 food matrices. Here we present a high sensitivity and high selectivity LC-MS/MS method that combines Multiple Reaction Monitoring (MRM) quantitation with MRM ratios as a first step of identification. Samples were re-analyzed using LC-MS/MS using information dependent acquisition (IDA) of QTRAP® MS/MS spectra. MS/MS spectra were searched against extensive mass spectral libraries for high confidence confirmation.
Food samples, including a variety of fruits and vegetables were extracted using a QuEChERS procedure and injected into LC-MS/MS after extensive dilution to minimize or possibly eliminate matrix effects. LC separation was performed using a Phenomenex core-shell Kinetex Biphenyl column and a gradient of water and methanol and ammonium formate buffer with a total run time of 15 min. Detection was performed on the AB SCIEX QTRAP® 6500 system using Electrospray Ionization (ESI). First injection was performed using the Scheduled MRM™ pro algorithm to reproducibly and accurately monitor 800 transitions for the quantitation and identification of 400 pesticides.
In a second injection already identified pesticides were confirmed based on MRM-IDA-MS/MS analysis. The acquisition of full scan MS/MS spectra helped to reduce false positive findings. Data processing was performed using MultiQuant and MasterView software.

P-110 Routine Targeted Quantitation and Identification of Pesticide Residues using Triple Quadrupole LC-MS/MS and Advanced Scheduling of MRM Transitions

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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 AB SCIEX triple quadrupole mass spectrometer 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-111 A New Fast and Sensitive HPLC-PDA Method for Analysis of Aflatoxins in Food Products that Eliminates the Need for Post-Column Derivitization

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Aflatoxins are secondary metabolites produced by Aspergillus Flavus and Aspergillus Paraciticus fungi that accumulate in agricultural foodstuffs often due to improper storage. These compounds are harmful or fatal to livestock and considered carcinogenic in both humans and animals. The levels of aflatoxins in many foodstuffs are regulated by many agencies including the US Food and Drug Administration and the European Union. Many currently accepted HPLC methods for aflatoxin analysis use post-column derivitization and fluorescence detection to reach the detection limits required by regulatory agencies. Although very sensitive, methods that require post column derivitization have significant peak dispersion and long runtimes. We have developed a complete method for analysis of aflatoxins in foodstuffs such as spices, grains and milk that is fast and sensitive while eliminating the need for derivitization. The analytes are first extracted and concentrated from the sample matrices using aflatoxin specific immunoaffinity SPE

columns. The samples are then quantified on a low dispersion HPLC system using solid core, superficially porous particle columns coupled with the new high sensitivity PerkinElmer PDA Plus detector. This results in fast runtimes (6 minutes injection-to-injections) with well resolved narrow peaks to obtain high sensitivity without derivitization.

P-112 Advanced Carbon Materials for Sample Preparation of Dioxins, and Furans from Complex Matrices

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Effectively cleaning matrix heavy samples like compost is a challenge in today's high though-
put laboratory setting. Samples should have as much interfering matrix removed as possible without affecting analyte recoveries to minimize instrument downtime. In this paper, we discuss new materials for use in the sample preparation section of EPA method 1613 concerning polychlorinated dibenzo-p-dioxins (PCDDs) and dibenzofurans (PCDFs). These classes of compounds are persistent organic pollutants that are routinely analyzed in meat, fish, food, and wastewater. This engineered carbon material enables higher throughput, better limits of detection, and less solvent usage.

Specifically, this work details the use of an engineered carbon adsorbent to streamline concentration and elution of PCDDs and PCDFs from waste sludge and other complex matrices. The material is produced by vapor depositing carbon on porous and mechanically stable silica. Increased recoveries were obtained using this new material vs the traditional cleanup using Celite(R)/CarbopackB . Furthermore, we demonstrate that significantly less toluene is necessary for full elution of the tetra through octa dioxins and furans when this adsorbent was used in place of Celite(R)/CarbopackB as described in EPA method 1613.

The carbon on silica materials greatly reduce interfering matrix, and we have demonstrated that it greatly extends column and liner lifetime in HRGC/MS. We observe a marked decrease in baseline noise and concomitant lowering of detection limits. We have also shown that use in a high throughput environment will extend the MS source cleaning interval, thus improving uptime and lowering costs. The material was packed in Fluorinated Ethylene Propylene (FEP) tubes with deactivated glass wool to eliminate the need for glassware, glassware autoclaving, and time consuming handling.

P-113 MS/MS^{ALL} with SWATH^{TM} Acquisition for Targeted and Untargeted Pesticide Screening

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The purpose of this work is to demonstrate a generic information-independent acquisition workflow to perform targeted and untargeted pesticide screening. Fruit and vegetable samples were extracted using QuEChERS approach, and then subjected to a generic RPLC separation. High resolution TOF MS data was acquired using a MS/MS^{ALL} with SWATH^{TM} (Sequential Windowed Acquisition of all Theoretical masses) method. Using this acquisition method, all ions that entered into mass spectrometer were fragmented regardless of what the precursor was. Compared to regular MS/MS^{ALL} approach, SWATH has much narrower Q1 selection window (typically 10 to 25 Da), and provides better selectivity and less comprehensiveness in post-acquisition data mining.
For targeted pesticide screening, high resolution extracted ion chromatograms (XIC) were obtained using characteristic MS/MS fragments for each analyte. The higher abundant fragment XIC was used for quantitation, while lower abundant XIC for qualification. Over 100 pesticides were analyzed using this approach. Due to the information-independent (untargeted) nature in acquisition, retrospective quantitation analysis maybe performed if any previously unknown pesticides become known.
For untargeted pesticide screening, MasterView^{TM} was employed to perform sample-control comparison and discover the possible pesticide ions. The "true" MS/MS spectra were then obtained by deconvolutional processing, and subjected to MS/MS library searching, empirical formula calculating and Chemspider database searching to tentatively determine the molecular formula and structure. A few untargeted pesticides were identified from non-organic samples by using their organic counterparts as control.

P-114 The Use of LC-MS/MS for the Analysis of Allergens in Foods

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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.

Here we present data acquired by LC-MS/MS for the screening and quantitation of multiple allergens including egg, milk, gluten, peanuts, tree nuts, sesame, and mustard.

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 methods utilizes *Scheduled* MRM™ and information dependant acquisition so that not only are multiple peptides detected for each allergen but full scan product ion data is collected at the same time for each peptide so that presence of allergen can be identified with high confidence.
In this presentation we will discuss how this approach compares against the traditional ELISA based assays on incurred samples. Selectivity, sensitivity, quantitative accuracy and repeatability of LC-MS/MS will be demonstrated. Latest method development includes the utilization of micro flow LC which was used to increase method throughput and save solvent costs.

P-115 Eliminating Matrix Effects and Interferences when Performing High Sensitivity and High Selectivity DMS-LC-MS/MS Pesticide Screening

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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. Matrix effects, like ion suppression and ion enhancement are a continuous challenge for food testing laboratories due to the complexity and variety of food samples to be tested. Here we present a high sensitivity and high selectivity LC-MS/MS method that combines quantitation with identification based on Multiple Reaction Monitoring (MRM) and full scan MS/MS data.

Food samples, including a variety of fruits and vegetables, but also tea, spices, and herbal products were extracted using a QuEChERS procedure and injected into LC-MS/MS after extensive dilution to minimize or possibly eliminate matrix effects. LC separation was performed using a Phenomenex core-shell Kinetex Biphenyl column and a gradient of water and methanol and ammonium formate buffer with a total run time of 15 min. Detection was performed on the AB SCIEX QTRAP® 6500 system using Electrospray Ionization. In addition, differential mobility separation (DMS) with SelexION™ technology was used to enhanced resolving power prior MS/MS detection to remove matrix interferences.

Over 400 targeted pesticides were quantified and identified using a Scheduled MRM method for best accuracy and reproducibility. The superior sensitivity of the MS/MS detector was used to dilute sample extracts extensively (up to 1000x) to completely eliminate matrix effects in most cases. DMS was used as an additional tool to remove matrix interferences when detecting tricky to analyze (small molecular weight and high polarity analytes).

P-116 The Quantitation of Mycotoxins in Cereals using a Simple Sample Extraction and LC-MS/MS using Fast Polarity Switching and MRM Scheduling

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Mycotoxins are produced by several strains of fungi both in the field, during storage, mixing and delivery of grain, human and animal food. Mycotoxins are known to be toxic and harm humans and animals as they are carcinogenic or otherwise cytotoxic and impair the immune system. Mycotoxins fall into several major classes and those which can affect the health of humans or animals include the aflatoxins, ochratoxins, Fusarium toxins, including fumonisins, zearalenone (ZON), trichothecenes, and ergot alkaloids.

Regulations for mycotoxin contamination for some of the major classes have been set in different countries. In the European Union the mycotoxin limits were harmonized in the regulation for contaminants in foodstuffs. Traditionally mycotoxin analyses have been carried out using multiple methods, each method just suitable for one single mycotoxin or a group of chemically similar compounds, e.g. aflatoxins. This has been due to the wide range of polarities and physical properties of these compounds.

Here we present a rapid, robust, sensitive and specific LC-MS/MS assay has been developed for the detection of several major classes of known toxic mycotoxins. The method uses a simple solvent extraction followed by a dilution and injection of extracts to achieve detection of mycotoxins below the regulatory requirements. Fast polarity switching and the Scheduled MRM™ algorithm were used with the AB SCIEX Triple Quad™ 5500 system to cover all mycotoxins of interest and to detect them with the best sensitivity, accuracy, and reproducibility. The presented method has been tested on several cereal based samples and has been shown to be robust enough to detect these toxins below the required limits and met European Legislation.

NOTES

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