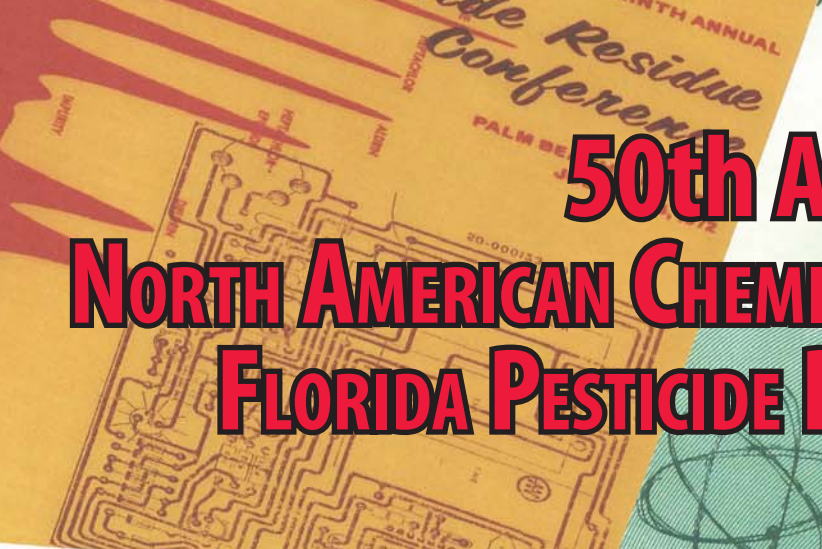
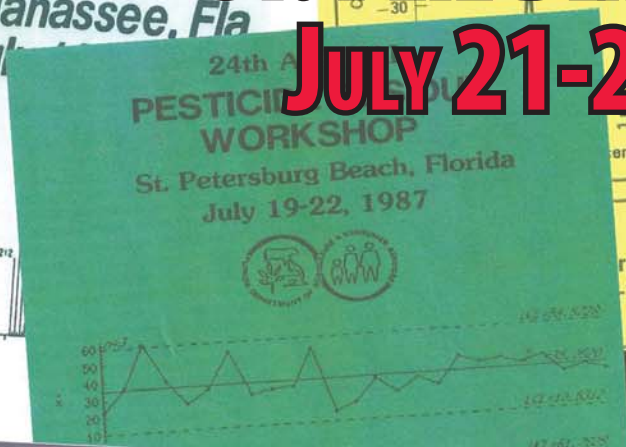
50th Annual
North American Chemical Residue Workshop
Florida Pesticide Residue Workshop

50th Year

TradeWinds Island Grand Resort
St. Pete Beach, Florida
July 21-24, 2013

"Bringing Scientists Together to Develop and Validate Better Methodologies"





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Chemical Residue Standards

- Pesticides and metabolites
- PCBs (all 209 congeners) & metabolites
- PBBs
- PBDEs (all 209 congeners) & metabolites
- Fluorinated PBDEs
- Other Brominated Flame Retardants
- α, β, γ - HBCDs
- Halo-dibenzo-p-dioxins and furans
- PAHs, nitro-PAHs, methyl-PAHs
- Explosives and metabolites
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FUTURE MEETING DATES

2014 July 20 - 23

2015 July 19 - 22

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

North American Chemical Residue Workshop Celebrates the 50th Anniversary of the FLORIDA PESTICIDE RESIDUE WORKSHOP

July 19, 2013

Dear Attendees and Exhibitors,

Welcome to the North American Chemical Residue Workshop. We extend our greetings to long-time attendees and first time attendees as well as our international guests. We also thank our Exhibitors and Sponsors for their generous support of the Workshop. Effective for 2013, FPRW will become the North American Chemical Residue Workshop. Although the name may change, the Organizing and Program Committees will continue to provide interesting scientific sessions and events that stimulate discussion and further our knowledge in chemical residue and the contaminant analysis.

This year's short course is "Chemical Residue Method Development Validation and Routine Analysis". The instructors are Richard Fussell and Simon Hird from the Food and Environment Research Agency, UK. This 2-day course provides introductory training in multi-residue and single-residue methods to determine pesticides and veterinary drugs in food and the environment. Emphasis is given to the critical steps required for successful method development, validation and implementation for residue monitoring and control. The course will include material on sample preparation and processing, extraction and clean up and the determination by GC-MS and LC-MS techniques. The training will also cover analytical quality control and validation of methods in accordance with relevant regulations and standards.

In addition to our welcoming reception on Sunday evening we have arranged for an unusual 50th Anniversary Celebration of the FPRW. This will take place on site in the Courtyard of the hotel and promises to be entertaining and a lot of fun. We hope you will join us in this celebration. Tickets are required and can be purchased at the registration desk.

Back by popular demand is the Mass Spec Forum on Tuesday afternoon featuring a moderated session focusing on issues associated with targeted and non-targeted methods. Bring your questions, ideas and concerns to discuss with colleagues in this interactive session!

We have a fantastic three day program lined up for you! Including sessions on:

Past, Present, and Future of Contaminant Analysis, Recent and Emerging Contaminant Issues, Veterinary Drug Analysis, Pesticide Registration Process, State-of-the-Art Tools in Contaminant Analysis, Forum on Mass Spectrometry, Updates from Government Labs, Cream of the Crop Technical Talks, and National and Global Regulatory Challenges.

Please visit the posters, speak to the authors and vote for the best poster. The Journal of Agricultural and Food Chemistry is again offering to publish a special edition affiliated with NACRW. If you have a manuscript to submit for publication, please inform Kate Mastovska, NACRW 2013 Program Chair.

Be sure to attend the Vendor workshops that start on Sunday evening and occur each day of the workshop. Check the schedule included in this booklet for dates times and locations. Please make sure to visit the Exhibition for one-on-one discussions with vendors. We want to thank all of the vendors for their participation and support for NACRW. Without them and those who are meeting sponsors we would not be able to have the entertainment as well as food and drink at our breaks. Please make an extra effort to visit their booths.

The NACRW Organizing Committee Planning Meeting takes place on Tuesday evening from 5:05 to 6:00 PM. NACRW is organized by volunteers who participate in the Organizing and Program Committees. Please join us for this year's meeting, get involved and help us continue an outstanding meeting. NACRW is sponsored by FLAG Works, Inc., a not-for-profit organization.

Finally, we would like to thank the 2013 Organizing Committee and the Program Committee and all of the volunteers for arranging and participating in a great meeting. Enjoy!

Michael Telepchak, Nina Huffstetler, Loretta Fourrier, Brad Barrett, Joe Romano (2013 Organizing Committee Officers)
Kate Mastovska, Paul Yang, Perry Martos, Sherri Turnipseed, Alaa Kamel, Lynda Podhorniak (2013 Program Committee Officers)

UCT

CONGRATULATIONS TO THE NACRW ON ITS 50TH YEAR!

UCT would like to welcome all the attendees to St. Pete Beach, FL and this year's North American Chemical Residue Workshop.

Come visit us at booths 40-41, and be sure to enjoy lunch on Tuesday July 23 in the Tarpon Room. Dr. Brian Kinsella will be presenting on the Chemistry of QuEChERS and focusing on newer applications and solutions.

UCT was the first company to create a commercial line of QuEChERS products for the analytical lab and we continue to be on the forefront of creating newer formats and technologies for the technique - including the Quick QuEChERS syringe cartridges pictured here and ChoroFiltr® product lines.

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MAJOR TOPIC AREAS WILL INCLUDE:

- Emerging Issues and Challenges
- Mode of Action and Resistance Management
- Discovery and Synthesis
- Agricultural Biotechnology
- Environmental Fate and Metabolism
- Ecological and Human Exposure and Risk Assessment
- Residues in Food and Feed
- Formulation and Application Technologies
- Stewardship, Regulation, and Outreach

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Brad Barrett, AB Sciex

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Paul Yang, Ontario Ministry of the Environment

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Sherri Turnipseed, USFDA

2011 Co-Chairs:
Alaa Kamel, USEPA
Lynda Podhorniak, USEPA

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Shawna Brown - Morse Laboratories
John Budin - Merieux NutriSciences
Jack Cochran - Restek
JoMarie Cook- FDACS
Tambra Dunams - CDC
Brian Eitzer - The Conn. Agr. Exp. Station
Shirley Elliott - Darling Analytical Laboratories
David Fries - University of Florida
Gale Hagood – Miss. State Chem Lab
Douglas Hayward - FDA
Simon Hird - FERA
Yelena Karaseva - FDA
Brian Kinsella - UCT
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André de Kok - NVWA
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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
POOL AREA
TAZ Activities
Pirate Island
Tennis Courts
The Pavilion
Parking
Cypress Villa
Parking
North Parking
EXHIBITS AND POSTERS
Lobby
REGISTRATION DESK AND MEETING ROOM
Magnolia Offices
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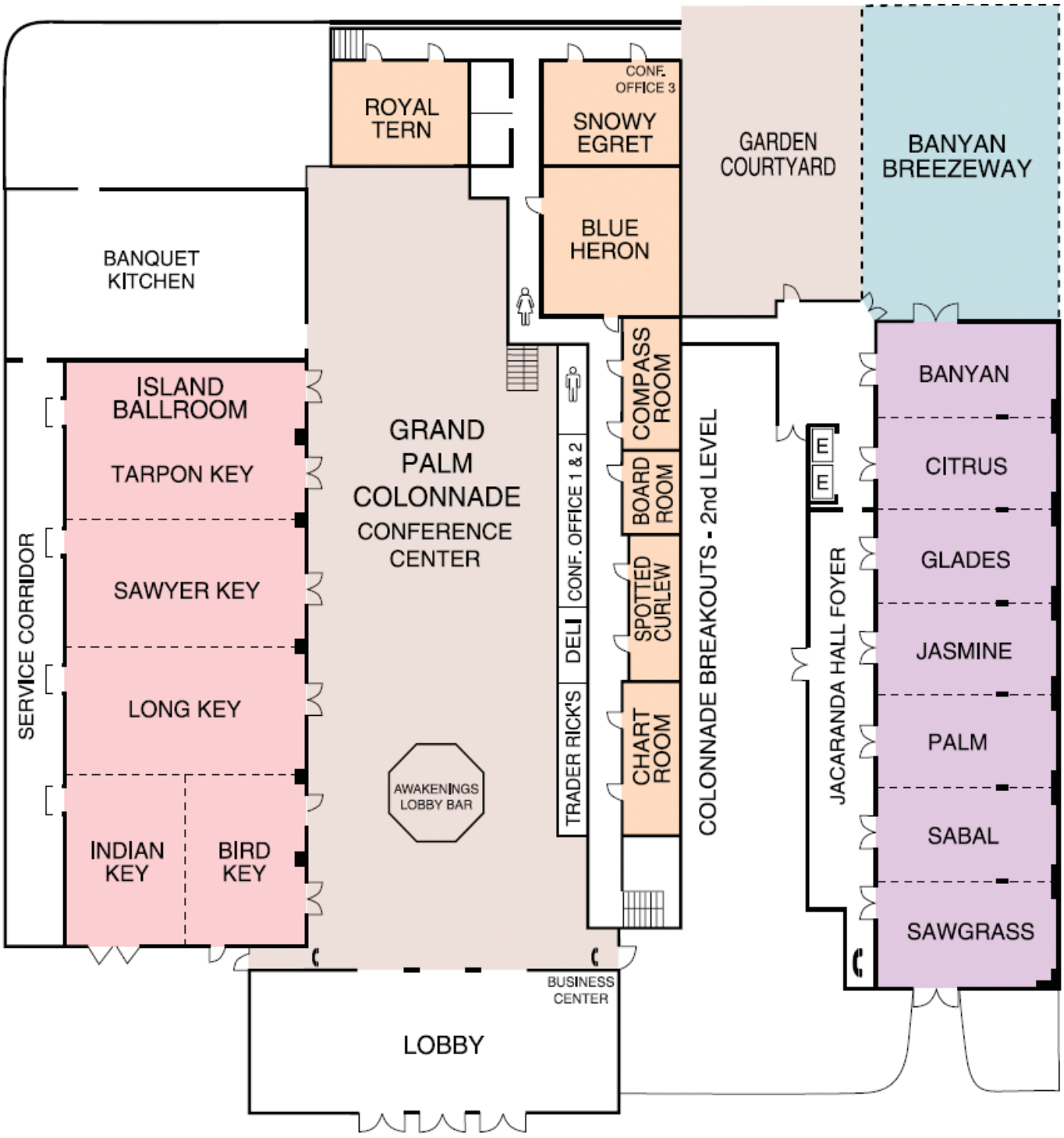

Technical Sessions: Long Key, Bird Key and Indian Key Ballrooms
Exhibits, Posters, Reception: The Pavilion
Vendor Seminars: Tarpon Key

We would like to thank the following companies for their support of the workshop!

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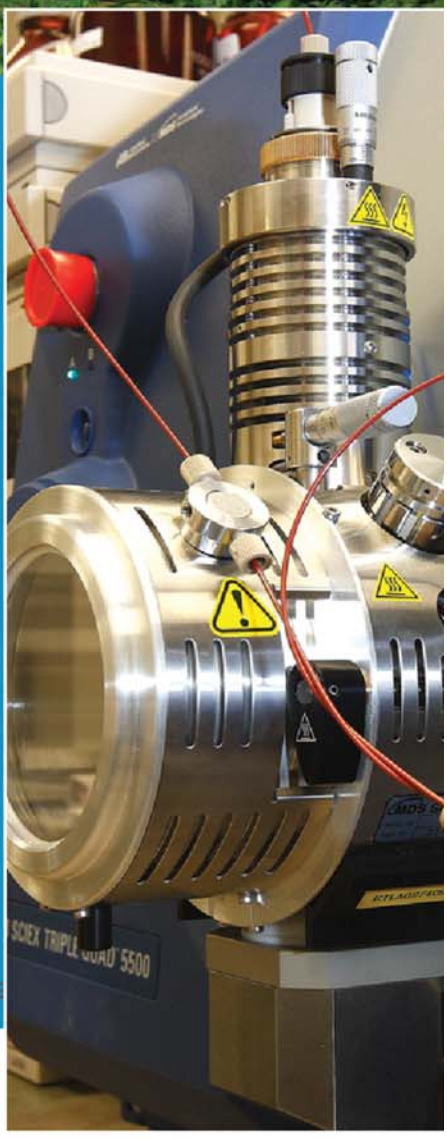

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50th Year

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MEETING AT A GLANCE

Saturday, July 20, 2013

8:00 am-4:00 pm	**Short Course: Part I - Richard Fussell and Simon Hird**	Snowy Egret

Sunday, July 21, 2013

8:00 am-3:00 pm	**Short Course: Part II - Richard Fussell and Simon Hird**	Snowy Egret
1:00 –5:00 pm	Exhibitor Setup	Pavilion
2:00 – 6:00 pm	Registration	Grand Palm Colonnade
4:00 – 6:00 pm	*Federal and State Labs Meeting*	
6:00 – 7:00 pm	**Restek Vendor Seminar**	**Tarpon Key**
7:00 – 9:00 pm	**Welcome Reception & Exhibition Opening**	**Pavilion**

Monday, July 22, 2013

All Day	Registration	Grand Palm Colonnade
7:00 – 10:00 am	Poster Board Set Up	Pavilion
7:15 – 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	**Past, Present, and Future of Contaminant Analysis**	Ballrooms
10:45 – noon	**Exhibition & Posters**	**Pavilion**
11:00 – noon	**Poster Session (authors for odd #s)**	**Pavilion**
12:15 – 1:15 pm	**Agilent Vendor Seminar**	**Tarpon Key**
1:30 – 3:10 pm	**Recent and Emerging Contaminant Issues**	Ballrooms
3:10 – 3:55 pm	**BREAK (Exhibition & Posters)**	**Pavilion**
3:55 – 5:05 pm	**Veterinary Drug Analysis**	Ballrooms
6:00 pm→	**Social Event: Celebration of 50th Anniversary**	**Banyan Breezeway/Garden Courtyard**

Tuesday July 23, 2013

All Day	Registration	Grand Palm Colonnade
All Day	**Exhibition & Posters**	**Pavilion**
7:15 – 8:15 am	**Early Morning Coffee**	**Grand Palm Colonnade**
7:15 – 8:15 am	**AB SCIEX Vendor Seminar**	**Tarpon Key**
8:30 – 10:30 am	**Pesticide Registration Process**	Ballrooms
10:30 – noon	**Exhibition & Posters**	**Pavilion**
11:00 – noon	**Poster Session (authors for even #s)**	**Pavilion**
12:15 – 1:15 pm	**UCT Vendor Seminar**	**Tarpon Key**
1:30 – 3:10 pm	**State-of-the-Art Tools in Contaminant Analysis**	Ballrooms
3:10 – 3:45 pm	**BREAK (Exhibition & Posters)**	**Pavilion**
3:45 – 5:00 pm	**Forum on Mass Spectrometry**	Ballrooms
5:05– 6:00 pm	**Organizing Committee Meeting**	Ballrooms
6:45 – 7:45 pm	**PerkinElmer Vendor Seminar**	**Tarpon Key**
8:00 pm →	Night Beach Volleyball Game	On the Beach

Wednesday, July 24, 2013

Until noon	Registration	Grand Palm Colonnade
Until noon	**Exhibition & Posters**	**Pavilion**
7:15 – 8:15 am	Early Morning Coffee	Grand Palm Colonnade
7:15 – 8:15 am	**Bruker Daltonics Vendor Seminar**	**Tarpon Key**
8:30 – 10:30 am	**Updates from Government Laboratories**	Ballrooms
10:30 – noon	**BREAK (Exhibition & Posters)**	**Pavilion**
12:15 – 1:15 pm	**Phenomenex Vendor Seminar**	**Tarpon Key**
1:30 – 3:10 pm	**Cream of the Crop Technical Talks**	Ballrooms
3:10 – 3:30 pm	**BREAK**	**Grand Palm Colonnade**
3:30 – 4:30 pm	**National and Global Regulatory Challenges**	Ballrooms
4:30 – 5:00 pm	**Closing (Poster Awards)**	Ballrooms
5:05 – 5:55 pm	**International Forum**	Ballrooms
6:00 – 7:00 pm	**Thermo Fisher Scientific Vendor Seminar**	Tarpon Key

Thursday, July 25, 2013

User Meetings

7:30 am – 9:30 am	AB Sciex User Meeting	Banyan Room
10:30 am – 12:30 pm	Agilent User Meeting	Citrus Room

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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 - 7:30 am – 4:30 pm
Wednesday - 7:30 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 11:00 am – noon on Monday
Poster Session B authors must be at their posters from 11:00 am – noon on Tuesday

Poster Prizes

Two poster prizes will be awarded of $100 each 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). **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 scrumptious food and an open bar 7:00 - 9:00 pm
Monday – 10:45 am - 5:00 pm
Tuesday - 7:30 am - 5:00 pm
Wednesday - 7:30 am – noon

Coffee and Breaks

Coffee will be available 7:15 - 8:15 am each morning in The Pavilion and the Grand Palm Colonnade. 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. 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 2013. Please inform Kate Mastovska (Katerina.Mastovska@covance.com), 2013 Program Chair, by September 3, 2013 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, 2013.

Copies of Presentations

Oral Presentations: Following the meeting, if 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 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 THE VOLUNTEERS!

The workshop would not be possible without your valuable assistance.

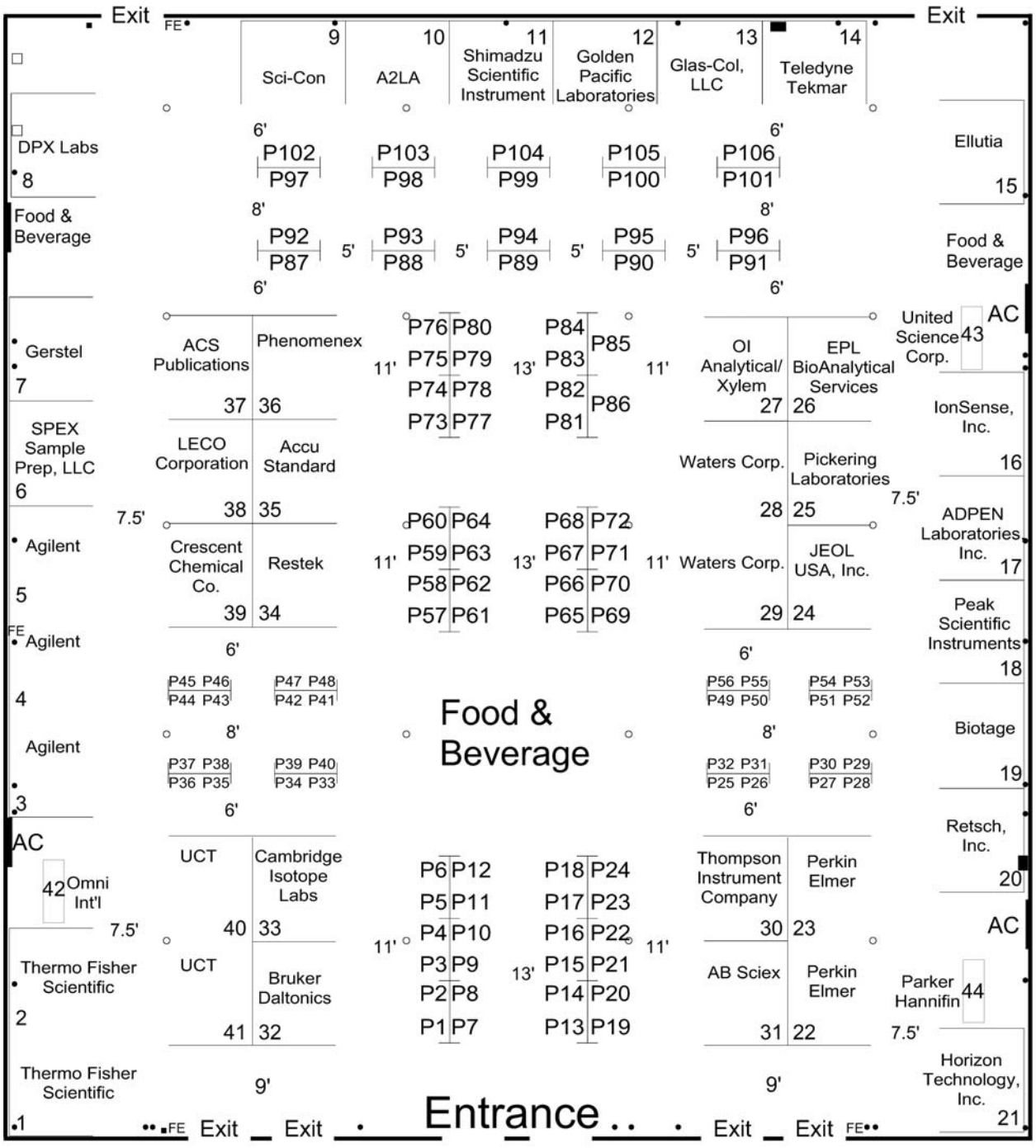
FUTURE MEETING DATES

2014 July 20 - 23
2015 July 19 - 22

All Meetings will be held at TradeWinds Island Grand Resort, St. Pete Beach, Florida

Exhibits and Poster Sessions

Location: Pavilion



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SHORT COURSE

Saturday, July 20, 2013 8:00 am to 4:00 pm
Sunday, July 21, 2013 8:00 am to 3:00 pm

Location: Snowy Egret

Chemical Residue Method Development, Validation and Routine Analysis

Instructors: Richard Fussell and Simon Hird

Food and Environment Research Agency, UK

This 2-day course provides introductory training in methods to determine pesticide and veterinary drug residues in food and the environment. The methods covered include multi-residue and single residue methods using mass spectrometric techniques. Emphasis is given to the critical steps required for successful method development, validation and implementation for residue monitoring and control. The course will include material on sample preparation and processing, extraction and clean-up and the determination by GC-MS and LC-MS techniques. The training will also cover analytical quality control and validation of methods in accordance with relevant regulations and standards.

REGISTRATION IS REQUIRED. The Short Course is full.

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VENDOR SEMINARS

Food and beverage provided by each company

Vendor Seminars: Please sign up at the meeting registration desk

V-1 Sunday Evening, July 21, 2013, 6:00 to 7:00 pm — Restek

Location: Tarpon Key

Are You a Gas Chromatography Mastermind?

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

Come test your gas chromatography skills and learn about alternatives to using helium carrier gas (e.g. hydrogen and nitrogen) for GC and GC-MS, large volume splitless injection without a PTV, fast pesticide analysis with split injection, and column trimming with method translation; these are techniques that save time and money for your lab. Take our quiz to win prizes and pick up an ultra-cool Chromatography Mastermind T-shirt, all while enjoying dinner and drinks with our famous NACRW Door Prize Host, Jonathan "Munch" Keim.

V-2 Monday Morning, July 22, 7:15 to 8:15 am — Waters Corporation

Location: Tarpon Key

A Novel Mass Screening Solution Based on Using UPLC-Qtof-MS and Automated Data Processing for the Detection of Pesticide Residues in Food

Richard J. Fussell, Food and Environment Research Agency, Sand Hutton, York, UK; richard.fussell@fera.gsi.gov.uk.

Pesticide residues remain high on the list of consumer concerns and thus, laboratories are required to screen samples for as many pesticides as possible in a single analysis within appropriate timescale and costs. This lecture will discuss the development and validation of a multi-residue qualitative screening method based on the use of a UPLC-high resolution mass spectrometer system (ACQUITY UPLC-I-Class coupled to a Xevo-G2-S-QToF-MS and UNIFI software) operated with non-targeted acquisition, but with MS^E to yield fragment ions. As outlined in EU guidelines (SANCO/12495/2011) the validation focused on the detectability of each of 190 pesticides at the screening detection limit (SDL) i.e. the concentration (typically around 0.01 mg/kg) at which a certain analyte will be detected in at least 95% of the samples.

The sample extraction was accomplished using the Waters DisQuE™ protocol based on the QuEChERS approach. UNIFI software, incorporating a 3D peak apex track integration algorithm, was used to automatically detect and filter peaks against a database containing the retention time, exact mass and fragment ions (where available) for more than 500 pesticides. The 'false negative/positive' rates, limitations and critical points will be discussed along with an interpretation of the results in the context of improving consumer confidence.

V-3 Monday Lunchtime, July 22, 12:15 to 1:15 pm — Agilent Technologies

Location: Tarpon Key

An Introduction to 'Target Deconvolution' – A Powerful New Feature of Agilent's Mass HunterQuantitative Software

Chris Sandy, Agilent Technologies UK Ltd., 610 Wharfedale Road, Winnersh, Berkshire, RG41 5TP, UK; chris_sandy@agilent.com

Target compound identification and quantitation using single quadrupole GC/MS systems has been used in routinely for many years. Target compound Identification is based on retention time matching and ion ratios of qualifying ions to a single quantification ion. Whilst this process works well for relatively 'clean' samples, more complex samples such as food or environmental extracts often present co-eluting matrix components that prevent accurate measurement of ion ratios. One approach to improve the quality of identification of target analytes using full scan GC/MS data

acquisition is to use mass spectral deconvolution and library searching. This facility has been available in Agilent's GC/MS Chemstation software for nearly 10 years and is known as Deconvolution Reporting Software (DRS). This presentation discusses the new 'Target Deconvolution' feature of the latest revision of Mass Hunter Quantitative software and shows it's implementation and use of Mass Hunter's powerful quantitative data review programs – 'Batch at a Glance' and 'Compounds at a Glance'.

Implementation of Modified QuEChERS Techniques for Modern Applications

Derick Lucas, Ph.D., Research Scientist, Chemical Supplies Division, Agilent Technologies, Wilmington, DE, USA

While improvements to instrument speed, selectivity, and sensitivity have reduced the amount of time spent on laboratory analysis, sample preparation has emerged as a common bottleneck for laboratory workflows. Routine analysis can include the investigation of hundreds of chemically diverse compounds in a variety of matrix types. The Quick, Easy, Cheap, Effective, Rugged, and Safe (QuEChERS) sample preparation method was developed as a solution to these analytical challenges. The QuEChERS method can decrease the amount of effort spent during sample preparation by providing fewer procedural steps, extraction of large groups of target compounds and method transferability to different commodities. This presentation will highlight examples that integrate modifications of the traditional QuEChERS method to support modern applications as they expand to new types of analytes and matrices.

V-4 Tuesday Morning, July 23, 2013, 7:15 to 8:15 am AB SCIEX

Location: Tarpon Key

Enhancing Routine Food Testing Beyond MRM: Using QTRAP and TripleTOF to Improve Sensitivity, Overcome Matrix Interferences, and Provide Better Confidence In Results

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

Routine food testing scientists face numerous challenges every day. They work hard to validate their results to ensure accuracy and prevent the reporting of false positive or false negative results. They are required to work with challenging food matrices that can create interferences and ambiguity in their results. And, they strive to achieve limits of detection that meet regulatory demands. This presentation will give an overview of how some of the unique functionalities of QTRAP and TripleTOF technology can help address some of these challenges and improve results for food testing labs above and beyond standard MRM acquisition.

V-5 Tuesday Lunchtime, July 23, 2013, 12:15 to 1:15 pm UCT

Location: Tarpon Key

The Chemistry of QuEChERS: Theory, Method Development and Applications

Brian Kinsella and Michael Telepchak, UCT, 2731 Bartram Road, Bristol, PA 19007

The QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe; pronounced "catchers") sample preparation approach was introduced in 2003 by Anastassiades *et al.* for the analysis of pesticide residues in fruits and vegetables. In QuEChERS, a high-moisture sample is extracted with an organic solvent in the presence of salts (and/or buffers) to induce phase separation. The organic phase is then subjected to dispersive solid-phase extraction (dSPE), which entails mixing sorbents with the extract to remove matrix co-extractives. The resulting supernatant can be analyzed directly or processed further if necessary. The approach is very flexible and since its inception several modifications have been made to the technique depending on analytes, matrices, instrumentation and analyst preferences. It has been widely adopted in pesticide residue analysis worldwide and has been successfully used to analyze hundreds of pesticides in a variety of foods. Due to its success, the technique has also been used in a variety of other analytical fields (e.g. veterinary drugs and environmental analysis). This presentation will outline the theory behind the QuEChERS approach. The attendee will gain an understanding of the different extraction procedures available (including buffered vs. unbuffered), and the variety of dSPE sorbents that can be used for clean-up. Advantages, pitfalls, method development and an outline of the wide variety of applications using QuEChERS will also be addressed.

V-6 Tuesday Evening, July 23, 2013, 7:00 to 8:00 pm PerkinElmer
Location: Tarpon Key

Accurate and rapid screening of 130 pesticides in lettuce using UHPLC-TOF
Jesse Hines, Senior LCMS Product Specialist, PerkinElmer, John's Creek, GA, USA

With the advent of large scale agricultural production, hundreds of pesticides have been synthesized in the last century and used widely to protect crops. Newer pesticides continue to be synthesized for crop usage which makes it important to analyze both targeted (or expected analytes) and non-targeted pesticides in food and in the environment. Against this backdrop, this session will focus on groundbreaking analytical tools and techniques applied to ensure the safety of our food supply.

V-7 Wednesday Morning, July 24, 2013, 7:15 to 8:15 am Bruker Dalltonics
Location: Tarpon Key

Faster Data-to-Report with Reduced Errors and Greater Data Integrity via Exception Based Reporting and PACER Software
Jim Edwards, Software Product Manager, Bruker Corporation 3500 West warren Avenue, Fremont CA 94538; jim.edwards@bruker.com

Today's analytical laboratory is constantly challenged with the generation of high-quality, legally defensible data in a rapid turnaround time from sample-in to report-out, all while maintaining a cost-competitive position. Instrumentation companies have seemingly focused on the hardware (data acqisition) rather than the software (processing and reporting) as the focus of innovation. Bruker has taken the step forward to adding innovation to its software platform to match the innovation in hardware. This talk will focus on the PACER platform showing how exception based reporting provides the better data integrity, reproducibility and speed to report for the laboratory. Through novel peak detection algorithms PACER virtually eliminates the need for time consuming and often arbitrary based re-integrations. Comparative demonstrations of the PACER software will be shown as part of the presentation.

V-8 Wednesday Lunchtime, July 24, 2013, 12:15 to 1:15 pm Phenomenex
Location: Tarpon Key

Recent Rapid Response Laboratory Projects: New Methods to Address Emerging Food Safety Issues Including the Recent Phenylbutazone Alert
Serena Lazzaro, Global Marketing Manager, Food Safety & Quality, Phenomenex, Torrance, CA ,USA and André Schreiber, Applications Manager, Food & Environmental Markets, AB SCIEX, Concord, ON, Canada

Recently the European Community was scandalized when it was discovered that horse meat had been illegally incorporated into food products. Concern was raised not only by the mislabelling of horse meat but that veterinary drugs such as phenylbutazone (Bute), might have entered the food chain. "Bute" a common veterinary analgesic is not approved for human use in most countries. This heightened the concern that the horse meat entered the food-chain, may have been contaminated with a banned drug. The recommended concentration proposed by the EU reference laboratory for NSAIDs is 5ppb (technical guidance CRL Guidance Paper, 2007).

A simple and fast method to confirm phenylbutazone residues in ground meat products at 5 µg/kg (ppb) was developed by the rapid response laboratory. The procedure uses a SPE cleanup with mixed-mode StrataX-A and a 5 minute LC/MS method utilizing core-shell Kinetex 2.6µ XB-C18 column for rapid throughput.

Phenomenex and ABSCIEX have evaluated potential interferences in optimizing LC resolving power and MS/MS identification using scheduled MRM and automated spectral library searching. These complete solutions are available as iMethod™ applications, where users can obtain a reproducible method and all the consumables required to quickly set up the analysis procedure in their laboratory.

This seminar will discuss multiple solutions including two screening methods for multi-class pesticides and antibiotic residues in different matrices and a confirmatory method for phenylbutazone in ground meat products.

V-9 Wednesday Evening, July 24, 2013, 6:00 to 7:00 pm **Thermo Fisher Scientific**
Location: Tarpon Key

Simplifying Productivity in Pesticides Analysis – The Very Latest Developments in GC and LC Triple Quadrupole MS Technology for Routine Screening and Quantifying Pesticides in Complex Samples

Paul Silcock, Thermo Fisher Scientific, Manchester, UK, and Dipankar Ghosh, Thermo Fisher Scientific, San Jose, CA

GC-MS/MS and LC-MS/MS are well established as the "routine workhorse" in many laboratories that are responsible for measuring pesticides 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 true 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 triple quadrupole mass spectrometry systems as well as complementary technologies that specifically address these needs.

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MEETING PROGRAM

Saturday, July 20, 2013

8:00 am-4:00 pm **Short Course: Part I** Snowy Egret
Chemical residue method development, validation and routine analysis
Richard Fussell & **Simon Hird** - Food and Environment Research Agency, UK

Sunday, July 21, 2013

8:00 am-3:00 pm **Short Course: Part II** Snowy Egret
Chemical residue method development, validation and routine analysis
Richard Fussell & **Simon Hird** - Food and Environment Research Agency, UK

1:00 –5:00 pm Exhibitor Setup Pavilion
4:00 – 6:00 pm *Federal and State Labs Meeting*
2:00 – 6:00 pm Registration Grand Palm Colonnade
6:15 pm → Technical Session Setup Ballrooms

6:00 – 7:00 pm **Restek Evening Seminar** **Tarpon Key**
V-1 **Are You a Gas Chromatography Mastermind?**
Jonathan Keim, Michelle Misselwitz, Julie Kowalski, and Jack Cochran;
Restek Corporation, Bellefonte, PA, USA

7:00 – 9:00 pm Welcome Reception Pavilion
Join us for scrumptious food and an open bar

Monday, July 22, 2013

All Day Registration Grand Palm Colonnade
7:00 –10:00 a.m. Poster Board Set Up Pavilion
10:45 am → Exhibition & Posters Pavilion
7:15 – 8:15 am Early Morning Coffee Grand Palm Colonnade

7:15 – 8:15 am **Waters Breakfast Seminar** **Tarpon Key**
V-2 **A Novel Mass Screening Solution Based on Using UPLC-Qtof-MS and Automated Data Processing for the Detection of Pesticide Residues in Food**
Richard Fussell, Food and Environment Research Agency, Sand Hutton, York, UK

8:30 – 10:45 am **Opening Oral Session:** **Ballrooms**
Past, Present, and Future of Contaminant Analysis
Moderator: Katerina Mastovska (Covance Laboratories, WI, USA)

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

8:50 – 9:15 am **George Fong** – Florida Dept. of Agriculture - retired, FL, USA
O-1 **On the 50th Anniversary of FPRW (Florida Pesticide Residue Workshop): A Forum of Residue Analysts in the Past Half Century**

9:20 – 9:45 am **David Klein** – Texas Tech University, TX, USA &
Marc Engel – Florida Dept. of Agriculture and Consumer Services, FL, USA
O-2 **FPRW Past, Present and Future**

9:50 – 10:15 am **Johannes Corley**, L. Kunkel, and Jerry J. Baron - IR-4 Headquarters, NJ, USA
O-3 **The IR-4 Project, 50 Years of Service to U.S. Growers and Consumers**

10:20 – 10:45 am **Yukiko Yamada** – Ministry of Agriculture, Forestry and Fisheries, Japan
O-4 **Japan's So-called "Positive List" of Pesticide and Veterinary Drug Residues, and Related Issues**

10:45 – noon Exhibition Opening Pavilion

11:00 – noon **Poster Session A:** Pavilion
Authors of odd poster numbers present
Poster Committee: Steven Lehotay, Jo Marie Cook, André de Kok, Brian Eitzer, Michael Kofel, and Sherri Turnipseed

12:15 – 1:15 pm **Agilent Technologies Lunch Seminar** **Tarpon Key**
V-3 **An Introduction to 'Target Deconvolution' – A Powerful New Feature of Agilent's Mass Hunter Quantitative Software**
Chris Sandy, Agilent Technologies UK Ltd, Winnersh, Berkshire, UK
and **Implementation of Modified QuEChERS Techniques for Modern Applications**
Derick Lucas, Agilent Technologies, Wilmington, DE, USA

1:30 – 3:10 pm **Oral Session 2:** **Ballrooms**
Recent and Emerging Contaminant Issues
Moderator: Steven Lehotay (USDA-ARS, PA, USA)

1:30 – 1:50 pm **Jana Hajslova**, Jana Pulkrabova, Kamila Kalachova, Ondrej Lacina, and Lucie Drabova – Institute of Chemical Technology, Czech Republic
O-5 **Analysis and Occurrence of Emerging Halogenated Contaminants in Food**

1:55 – 2:15 pm **Thierry Delatour** – Nestlé Research Centre, Switzerland
O-6 **Analytical Methods to Address Chemical Emerging Issues from Early Management to Crisis Situation: An Industrial Perspective**

2:20 – 2:40 pm **Richard Fussell**, Monica Garcia-Lopez, David Mortimer, Stuart Wright, Monika Sehnalova, Chris Sinclair, Alwyn Fernades, and Matthew Sharman – Food and Environment Research Agency, UK and Food Standards Agency, UK
O-7 **An investigation into the Potential Uptake of Environmental Contaminants into Food: Real Scenarios**

2:45 – 3:05 pm **Michelangelo Anastassiades**, Julia Hepperle, Daniela Roux, and Anja Barth – EU Reference Laboratory for Pesticide Residues requiring Single Residue Methods, Chemischen und Veterinäruntersuchungsämter (CVUA), Germany
O-8 **Factors Affecting the Extractability of Incurred Pesticides**

3:10 – 3:55 pm **BREAK** (Exhibition & Posters) Pavilion

3:55 – 5:05 pm **Oral Session 3:** **Ballrooms**
Veterinary Drug Analysis
Moderator: Perry Martos (University of Guelph, ON, Canada)

3:55 – 4:15 pm **Steven Lehotay** and Alan Lightfield – Agricultural Research Service, USDA, PA, USA
O-9 **Assessment of False Positives and False Negatives in the Multiclass, Multiresidue Analysis of Veterinary Drugs in Animal Tissues by UHPLC-MS/MS**

4:20 – 4:40 pm **Simon Hird,** Jonathan Guest, Richard Ginn and George Stubbings – Food and Environment Research Agency, UK
O-10 **Implementing Multi-Residue Methodologies for Monitoring Residues in Foods using LC-MS(/MS)**

4:45 – 5:05 pm **Mary Carson**, Sean Conklin, Nohora Shockey, Kevin Kubachka, Karyn Howard – US FDA Center for Veterinary Medicine, US FDA Center for Food Safety and Applied Nutrition, and US FDA Forensic Chemistry Center, USA
O-11 **Development of an IC-ICP-MS Method to Determine Inorganic Arsenic in Liver from Chickens Treated with Roxarsone**

6:00 pm→ Social Event Celebration of 50th Anniversary Banyan Breezeway/Garden Courtyard
Tickets are required and may be purchased at the Registration Desk

Tuesday, July 23, 2013

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

7:15 – 8:15 am **AB SCIEX Breakfast Seminar** **Tarpon Key**
V-4 **Enhancing Routine Food Testing Beyond MRM: Using QTRAP and TripleTOF to Improve Sensitivity, Overcome Matrix Interferences, and Provide Better Confidence in Results**
André Schreiber, AB SCIEX, Concord, ON, Canada

8:30 – 10:30 am **Oral Session 4:** Ballrooms
Pesticide Registration Process – Panel Discussion
Moderator: Johannes Corley (IR-4, NJ, USA)

O-12
(1) New Pesticide Development – **Lesley Czochor** (DuPont, DE, USA)
(2) Pesticide Registration Studies – **Thomas Gould** (Bayer CropScience, USA)
(3) Field Trials and Crop Grouping – **Johannes Corley** (IR-4, NJ, USA)
(4) Pesticide Tolerance Setting in the USA – **Michael Doherty** (US EPA, USA)
(5) Codex MRL Setting and Harmonization – **Yukiko Yamada** (MAFF, Japan; JMPR)

10:30 – noon Exhibition & Posters Pavilion

11:00 – noon **Poster Session B:** Pavilion
Authors of even poster numbers present
Poster Committee: Steven Lehotay, Jo Marie Cook, André de Kok, Brian Eitzer, Michael Kofel, and Sherri Turnipseed

12:15 – 1:15 pm **UCT Lunch Seminar** **Tarpon Key**
V-5 **The Chemistry of QuEChERS: Theory, Method Development and Applications**
Brian Kinsella and Michael Telepchak, UCT, Bristol, PA, USA

1:30 – 3:10 pm **Oral Session 5:** **Ballrooms**
State-of-the-Art Tools in Contaminant Analysis
Moderator: Brad Barrett (AB Sciex, MA, USA)

1:30 – 1:50 pm **Perry Martos** and Nick Schrier – University of Guelph, ON, Canada
O-13 **Identification of Unexpected Contaminants in Veterinary Practice**

1:55 – 2:15 pm **Hans Mol**, Ruud van Dam, Paul Zomer – RIKILT – Institute of Food Safety, The Netherlands
O-14 **Direct MS Detection of Residues, Contaminants and Adulterants in the Food Chain**

2:20 – 2:40 pm **Sherri Turnipseed**, Jack Lohne, Wendy Andersen, Joseph Storey, Susan Young, Justin Carr, and Mark Madson – US FDA Denver Federal Center and Denver Laboratory, CO, USA
O-15 **Challenges in Implementing HRMS Screening for Veterinary Drug Residues in a Regulatory Laboratory**

2:45 – 3:05 pm **Mark Crosswhite**, Ghislain Gerard, and Walter Hammack – Florida Dept. of Agriculture and Consumer Services, FL, USA
O-16 **Triumphs and Challenges of High-Resolution Mass Spectrometry in Comprehensive Pesticide Residue Screens**

3:10 – 3:45 pm **BREAK** (Exhibition & Posters) Pavilion

3:45 – 5:00 pm **Forum on Mass Spectrometry** **Ballrooms**
Moderator: Walter Hammack (Florida Dept. of Agriculture and Consumer Services, FL, USA)

5:05– 6:00 pm **Organizing Committee Meeting** Ballrooms

6:45 – 7:45 pm **Perkin Elmer Evening Seminar** **Tarpon Key**
V-6 **Accurate and Rapid Screening of 130 Pesticides in Lettuce using UHPLC-TOF**
Jesse Hines, Perkin Elmer, John's Creek, GA, USA

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

Wednesday, July 24, 2013

Until noon Registration Grand Palm Colonnade
Until noon Exhibition & Posters Pavilion
7:15 – 8:15 am Early Morning Coffee Grand Palm Colonnade

7:15 – 8:15 am **Bruker Corporation Breakfast Seminar** **Tarpon Key**
V-7 **Faster Data-to-Report with Reduced Errors and Greater Data Integrity via Exception Based Reporting and PACER Software**
Jim Edwards, Bruker Corporation, Fremont, CA, USA

8:30 – 10:30 am **Oral Session 6:** **Ballrooms**
Updates from Government Laboratories
Moderator: Sherry Garris (South Carolina Dept. of Agriculture, SC, USA)

8:30 – 9:00 am **Jo Marie Cook** – Florida Dept. of Agriculture and Consumer Services, FL, USA
O-17 **Florida's 2013 Chemical Residue Program in Review**

9:00 – 9:20 am **Brian Eitzer**, Walter Krol, Terri Arsenault, Jason White, Ellen Sloan – The Connecticut Agricultural Experiment Station and The Connecticut Department of Consumer Protection, CT, USA
O-18 **Pesticide Residues in Produce: The Connecticut Market Basket Survey**

9:20 – 9:40 am **Jona Verreth**, Angela Schaner, Heidi Hickes – Montana Department of Agriculture, MT, USA
O-19 **Montana's Program for Monitoring Trace Level Pesticides in Groundwater**

9:40 – 10:00 am **Ping Wan** – Office of Indiana State Chemist, IN, USA
O-20 **2012 Indiana's Bee Kill Investigations: Field observations, sampling and pesticide residue analysis**

10:00 – 10:05 am **Yoko Johnson** – Minnesota Department of Agriculture, MN, USA
Introduction of a New Information Resource for Pesticide Residue Chemists

10:05 – 10:30am **Panel discussion**

10:30 – noon **BREAK** (Exhibition & Posters) Pavilion

12:15 – 1:15 pm **Phenomenex Lunch Seminar** **Tarpon Key**
V-8 **Recent Rapid Response Laboratory Projects: New Methods to Address Emerging Food Safety Issues Including the Recent Phenylbutazone Alert**
Serena Lazzaro, Phenomenex, Torrance, CA ,USA and André Schreiber, AB SCIEX, Concord, ON, Canada

1:30 – 3:10 pm **Oral Session 7:** **Ballrooms**
Cream of the Crop Technical Talks
Moderator: Brian Eitzer (The Connecticut Agricultural Experiment Station, CT, USA)

1:30 – 1:50 pm **Robert Sheridan**, Jenifer Kemnah, Kristen Hafler, and Thomas Tarantelli – New York State Department of Agriculture and Markets, NY, USA
O-21 **Two Current Topics of Food and Feed Contamination: Identification and Quantification of 6 Illegal Antibiotics in Chinese Chicken Jerky Dog Treats and the Identification of Illegal Colors in Various Food Products**

1:55 – 2:15 pm **Robert Voyksner**, Jerry Zweigenbaum, and Jennifer Sealey-Voyksner – LCMS Limited, NC, USA and Agilent Technologies, DE, USA
O-22 **Moving Food Safety to Peptides: New Approaches using LC-MS for the Detection of Plant Allergens**

2:20 – 2:40 pm **Anthony Macherone** – John Hopkins University, MD, and Agilent technologies, DE, USA
O-23 **Environmental Analysis and the Exposome Phase I: Chemical Cartography using GC/Q-TOF Mass Spectrometry**

2:45 – 3:05 pm **Marc Engel** – Florida Dept. of Agriculture and Consumer Services, FL, USA
O-24 **Can Oyster Harvest Areas Be Identified By Their Cadmium- Lead Signature?**

3:10 – 3:30 pm **BREAK** **Grand Palm Colonade**

3:30 – 4:30 pm **Oral Session 8:** **Ballrooms**
National and Global Regulatory Challenges
Moderator: Katerina Mastovska (Covance Laboratories, WI, USA)

3:30 – 3:55 pm **Sarah McMullen** and Carl Sciacchitano – US FDA, Office of International Programs, MD, USA
O-25 **Moving Forward Together To Enhance Global Product Safety: A Laboratory Perspective**

4:00 – 4:25 pm **David Acheson** – Leavitt Partners, UT, USA
O-26 **Understanding the Food Safety Modernization Act (FSMA) in Relation to Chemical Risks**

4:30 – 5:00 pm **Closing (Poster Award)** **Ballrooms**

5:05 – 5:55 pm **International Forum** **Ballrooms**
Discussion forum on FSMA and international harmonization
Organizers: Sarah McMullen and Carl Sciacchitano, US FDA

6:00 – 7:00 pm **Thermo Fisher Scientific Evening Seminar** **Tarpon Key**
V-9 **Simplifying Productivity in Pesticides Analysis – The Very Latest Developments in GC and LC Triple Quadrupole MS Technology for Routine Screening and Quantifying Pesticides in Complex Samples**
Paul Silcock, Thermo Fisher Scientific, Manchester, UK and Dipankar Ghosh, Thermo Fisher Scientific, San Jose, CA

Thursday, July 25, 2013

User Meetings:

7:30 am – 9:30 am	**AB Sciex User Meeting**	Banyan Room
10:30 am – 12:30 pm	**Agilent Technologies User Meeting**	Citrus Room

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POSTERS

Session A (ODD NUMBERED POSTERS P1, P3, P5, etc.)
Authors stand by their posters from 11:00 am – noon on Monday

Session B (EVEN NUMBERED POSTERS P2, P4, P6, etc.)
Authors stand by their posters from 11:00 am-noon on Tuesday

P1 **What Pesticides Have You Eaten Today?**
Liesl Krone, Action Middle School and André Schreiber, AB SCIEX, Concord, ON, Canada

P2 **Cell Culture Studies of Exposure Response: Highlighting Biochemical Pathway Changes**
Robert Trengove et al.; Murdoch University, Murdoch, WA, USA

P3 **Development and Validation of a Multi-Residue Method for the Determination of 340 Pesticides in Agricultural Products using the GC-MS/MS Triple Quadrupole Technique**
André de Kok and Barbara Kiedrowska, NVWA - Netherlands Food and Consumer Product Safety Authority, Wageningen, The Netherlands

P4 **The Art of Method Development: Modification of an Analytical Method for the Analysis of a Clopyralid in Animal Tissues and the Pitfalls Encountered.**
Stanley R. Shaffer, et al.; Analytical Bio-Chemistry Laboratories, Columbia, MO, USA

P5 **Pesticide and Veterinary Drug Residue Analysis by Multiple Walled Carbon Nanotubes (MWCNTs) and multi-Plug-Filtration-Cleanup (m-PFC) Method**
Canping Pan, et al. Department of Applied Chemistry, China Agricultural University, Beijing, China

P6 **Laser Diode Thermal Desorption Mass Spectrometry for Veterinary Drug Residue Analysis**
Wendy C. Andersen, et al. Animal Drugs Research Center, U.S. Food and Drug Administration, Denver, CO, USA

P7 **Analysis of Multiclass Veterinary Drug Residues in Baby Food by Ultra Fast Chromotagraphy with High Performance Triple Quadropole Mass Spectormetry**
Charles Yang, et al. Thermo Fisher Scientific, San Jose, CA, USA

P8 **Analysis of Targeted and Non-Targeted Identified Contaminants in Storm Water Retention Ponds using LC-HRMS with Online Solid Phase Extraction**
Jennifer Massi, et al. Thermo Fisher Scientific, San Jose, CA, USA

P9 **Identification of illegal colors and dyes in various food products by LC/MS/MS**
Robert Sheridan and Thomas Tarantelli, New York State Department of Agriculture and Markets, Albany, NY, USA

P10 **Identification and quantification of 6 illegal antibiotics in Chinese chicken Jerky dog treats**
Robert Sheridan, et al. New York State Department of Agriculture and Markets, Albany, NY, USA

P11 **Identification Criteria for Residues Determined by LC-MS/MS: Are They Fit-for-Purpose?**
Hans Mol, et al. RIKILT – Institute of Food Safety, Wageningen, Netherlands

P12 **Validation of a Qualitative Screening Method for Pesticides in Fruits and Vegetables by GC-(APCI)QTOF-MS**
Tania Portoles, et al. Research Institute for Pesticides and Waters, University Jaumel, Castellón, Spain;

P13 **A Miniaturized Residue Analytical Method for the Determination of Zoxamide and its Two Acid Metabolites in Ginseng Using LC-MS/MS**
Lynda Podhorniak, U.S. EPA Analytical Chemistry Laboratory, Fort Meade, MD, USA

P14 **Multi-Residue Method for the Analysis of >140 Pesticide Residues in Fish using Fast, Low-Pressure GC-MS/MS**
Yelena Sapozhnikova, USDA Agricultural Research Service, Wyndmoor, PA, USA

P15 **Evaluation of Pesticides Contamination in Guarani Aquifer Recharge Area: GIS as Tool for Area Selection and Validation of Chromatographic Method**
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Tyler Trent, Teledyne Tekmar, Mason, Ohio, USA

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Kory Kelly and Matthew Trass, Phenomenex, Torrance, CA 90501, USA

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Matthew Trass, et al. Phenomenex,Torrance, CA, USA

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Sarah E. McMullen, et al. US Food and Drug Administration, Atlanta, GA, USA

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Jia Wang, et al. Thermo Fisher Scientific, San Jose, CA 95134, USA

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Eugene Chang, et al. US FDA Pacific Regional Laboratory Southwest, Irvine, CA, USA

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Johannes Corley, et al. IR-4 Headquarters, Rutgers, The State University of New Jersey, Princeton, NJ, USA

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George F. Antonious, et al. Kentucky State University, College of Agriculture, Frankfort, KY, USA

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Tho Pham, et al. Eurofins Analytical Laboratories, Metairie, LA, USA

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Masaaki Ubukata, et al. JEOL USA, Inc., Peabody, MA, USA

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Steven Winter and Raymond Allum, Florida Department of Agriculture and Consumer Services, Tallahassee, FL, USA

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Limian Zhao and Mike Szelewski, Agilent Technologies Inc., Wilmington, DE, USA

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Narong Chamkasem and Tiffany Harmon, US FDA Southeast Regional Laboratory, Atlanta, GA, USA

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Paul Silcock, et al. Thermo Fisher Scientific, Austin, TX, USA

P101 **Optimization of Simultaneous Derivatization for Rapidly Screening Banned Anabolic Steroids in Dietary Supplements by GC-MS-MS**
Jason Tang and Timothy Baker, NSF International, Analytical Chemistry Laboratory, Ann Arbor, MI, USA

P102 **A Simple Solution for Multi-Residue Analysis in Vegetables from Sample Prep to GC-MS/MS and LC-MS/MS Screening**
Helen Sun, et al. Bruker Daltonics, Inc., Fremont, CA, USA

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Ed George, Bruker Daltonics, Inc., Fremont, CA, USA

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Zicheng Yang, et al. Bruker Daltonics, Inc., Fremont, CA, USA

P105 **Analysis of Organophosphorus Pesticides in Baby Foods Using a Triple-Quadrupole GC/MS/MS**
Laura Chambers, et al. Shimadzu Scientific Instruments, Columbia, MD, USA

P106 **A Quick Assay for the Quantitation of Deoxynivalenol in Grain Samples by Liquid Chromatography with UV Detection**
Victor A. Vega, et al. US Food and Drug Administration, Southeast Regional Laboratory, Atlanta, GA, USA

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ORAL PRESENTATION ABSTRACTS

O-1 On the 50th Anniversary of FPRW (Florida Pesticide Residue Workshop): A Forum of Residue Analysts in the Past Half Century

George Fong

Florida Department of Agriculture *(retired)*, Tallahassee, FL, USA; Gandwfong@comcast.net

The forum began with analysts' quest of how to do the job. As further into the work, pesticide residues in food and in the environment were on the national agenda. Residue analysts were facing challenges of detecting more chemicals at lower concentrations and with accountability. FPRW provides the opportunity for net-working in sharing knowledge, problems and interests. In doing so, friendship developed among colleagues.

O-2 FPRW Past, Present and Future

David Klein[1] & Marc E. Engel [2]

[1]**Texas Tech University**, Department of Environmental Toxicology, PO Box 41163, Lubbock, TX 79409, USA; david.klein@ttu.edu
[2]**FL Dept. Of Agriculture and Consumer Services,** 3125 Connor Blvd #9, Tallahassee, FL 32399, USA; marc.engel@freshfromflorida.com

During its fifty years of existence, FPRW/NACRW has been a great opportunity for science, friendship and fun. The last 20 years has seen remarkable changes in this conference from its evolution from FPRW to NACRW to its growth in international attendees and speakers. But perhaps the greatest evolution was the introduction and implementation of mass spectrometry (MS) beyond gas chromatography with single quadrupole (GC-MS). In the early 1990's GC ion traps (GC-IT) started the evolution and the evolution continues today with the move to liquid chromatography - high-resolution mass spectrometry (LC-HRMS) techniques. This talk will focus on the years 1992-2000 with a special emphasis on George Fong and his leadership of FPRW, the evolution of MS and the FUN!!!! shared with colleagues during this time.

O-3 The IR-4 Project, 50 Years of Service to U.S. Growers and Consumers

Johannes Corley, Daniel L. Kunkel, and Jerry J. Baron

IR-4 Headquarters, Rutgers, The State University of New Jersey, 500 College Rd. East # 201 W., Princeton, NJ 08540, USA; jcorley@aesop.rutgers.edu

In 1963, the U.S. established the IR-4 project to facilitate the registration of pest management tools for specialty crops. Since then, IR-4 has been assisting U.S. producers of fruits, vegetables and other specialty crops manage their pest problems by providing safer and more effective chemical and biological mechanisms for insect, weed and disease control. Fifty years and more than 26,000 crop uses later, the IR-4 project still thrives and continues tackling even more challenging issues facing specialty crop growers with greater focus and vigor than ever. Today, several nations around the world are starting to emulate the IR-4 program in their own countries. A time-lapse view of the IR-4 project's efforts over the last 50 years keeping the U.S. food supply safe and plentiful and enabling you, the consumer to have plenty of delicious and nutritious fruits and vegetables on your table, will be presented.

O-4 Japan's So-called "Positive List" of Pesticide and Veterinary Drug Residues, and Related Issues

Yukiko Yamada

Ministry of Agriculture, Forestry and Fisheries, 1-2-1 Kasumigaseki, Chiyoda-ku, Tokyo 100-8950, Japan; yukiko_yamada@nm.maff.go.jp

In Japan, chemical residues are controlled by the Food Sanitation Law. This law stipulates maximum residue limits (MRL) for pesticide and veterinary drugs and maximum levels (ML) for contaminants. In response to concerns of the public about food safety, the Ministry of Health, Labour and Welfare of Japan (MHLW), bearing authority and responsibility for the law, introduced a positive list approach to the regulation of pesticide and veterinary drug residues in foods. The "Positive List" contains MRLs for a total of more than 700 pesticides and veterinary drugs including feed additives. It is called "positive list" not because of indicating chemicals

approved for use but chemical/commodity combinations to be analyzed for compliance. If specific chemical is not shown on the list, its residue concentration shall be at or lower than 0.01 mg/kg. In developing the list, MHLW fully uses Codex MRLs whenever they exist and prove to protect the health of consumers after the exposure assessment. When a chemical is registered/approved in Japan without Codex MRL, the respective national MRLs are used. For those chemicals not registered/approved in Japan or considered by Codex, MRLs established in Australia, Canada, EU, New Zealand and USA are used as reference. For any chemical, dietary exposure is estimated and compared with its ADI. For contaminants, previously MLs were set by allocating certain portions of toxicological end point such as TDI to certain commodities. Now, they are set by applying the ALARA principle to occurrence data of contaminants.

O-5 Analysis and Occurrence of Emerging Halogenated Contaminants in Food

Jana Hajslova, Jana Pulkrabova, Kamila Kalachova, Ondrej Lacina, and Lucie Drabova

Institute of Chemical Technology, Technicka 3, 16628 Prague, Czech Republic; jana.hajslova@vscht.cz

A wide range of contaminants has emerged in the recent decade on the target list of laboratories responsible for food safety control. Among them, brominated flame retardants (BFRs) / their metabolites and polyfluorinated alkyl substances (PFAS) have become of a high concern. In this presentation, high throughput analytical strategies enabling reliable analysis of (ultra)trace levels of these two groups of halogenated compounds will be presented. Using QuEChERS-like procedure for fish/seafood sample processing followed by gas chromatography coupled with mass spectrometry (GC-MS), integration into a single run BFRs analysis together with other pollutants, such as chlorinated persistent organic pollutants (POPs) and polycyclic aromatic hydrocarbons (PAHs) potentially occurring in fish/seafood has been shown to be feasible. Performance characteristics achievable by various types of recent, cutting-edge mass analyzers will be critically assessed. Regarding PFAS, attention will be mainly paid to the analysis of polyfluorinated surfactants (PFS) including polyfluoro alkyl phosphates (PAPS), which are precursors of various persistent fluorine-containing contaminants. Besides of a new ultrahigh performance liquid chromatography - tandem mass spectrometry (U-HPLC-MS/MS)- based method developed for their analysis in food contact materials (from which they are transferred into food), also challenges offered by high definition mass spectrometry (HDMS) employing ion mobility MS will be demonstrated.

O-6 Analytical Methods to Address Chemical Emerging Issues from Early Management to Crisis Situation: An Industrial Perspective

Thierry Delatour

Nestlé Research Centre, Vers-chez-les-Blanc, 1000 Lausanne 26, Switzerland; thierry.delatour@rdls.nestle.com

In food industry, analytical methods are key-components of product quality as they are fully integrated in the decision-making process for demonstrating safety and compliance. On an operational standpoint, they help to ensure that raw material composition fits with the specifications, and also to demonstrate the absence of any deviation along the manufacturing process. In research and development, methods participate in the innovation stream by making sure future products are nutritionally adapted, free of any safety risk, and able to deliver the claim-specific benefit.

Progress in food science and events related to worldwide food trade, unexpected climatic conditions or fraud may trigger unusual situations for food production, leading to potential risks for consumers, either in terms of safety or food supply, and environment. The successful identification of risks at their early inception is at heart of public health and environmental protection; this is possible by supporting the development, establishment and operation of structures for the screening and analysis of information sources with a view to identify emerging risks. In such a process, analytical methods participate in generating precise and factual information, useful to deploy suitable action plans for early management.

If inadequate attention is paid to indicators or signals, issues may evolve into crisis, with high impact on consumer trust towards the food production and its supply chain. Depending on the public perception regarding the damage, not only a single brand is impacted but also the entire food sector. Here again, analytical data obtained with recognized methods actively contribute to restore the confidence at authority bodies, and ultimately consumers. The contribution of research and development for early warning and management of issues will be presented, and the case of melamine will be shown for illustrating the role of analytical sciences in facing major challenges in a crisis situation.

O-7 An Investigation into the Potential Uptake of Environmental Contaminants into Food; Real Scenarios

Richard J. Fussell[1], Monica Garcia-Lopez[1], David N Mortimer[2], Stuart Wright, Monika Sehnalova[1], Chris Sinclair[1], Alwyn Fernades[1], and Matthew Sharman[1]

[1] **Food and Environment Research Agency**, Sand Hutton, York, YO41 1LZ, UK; richard.fussell@fera.gsi.gov.uk
[2] Food standards Agency, 3B Aviation House, 125 Kingsway, London WC2B 6NH, UK

Human exposure to emerging contaminants by indirect routes is of increasing interest. It is now understood that some groups of compounds, previously not considered as a risk, may enter the environment and subsequently the food chain by various pathways during their production, usage or disposal. Therefore, a research project has been undertaken to assess the potential for the contamination of food by selected human pharmaceuticals (HPs), veterinary medicines (VMs) and personal care products (PCPs). The first phase was prioritisation of those HPs, VMs and PCPs that might be of greatest concern regarding human health if they are present in food. The prioritisation considered many factors including usage toxicology, persistence, uptake and bioaccumulation potential, results from previous prioritisation exercises and reported environmental occurrence. The second phase involved the development and validation of suitable multi-class multi-residue methods for the analysis of the priority contaminants at low ng/g concentrations. The validated methods were then used for the analysis of samples of mushrooms, vegetables, fodder crops, aquaculture products and animal tissues produced in scenarios identified as a potential risk of contamination. The background to the study along with details of some of the analytical challenges and preliminary results will be discussed.

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O-8 Factors Affecting the Extractability of Incurred Pesticide Residues

Michelangelo Anastassiades, Julia Hepperle, Daniela Roux, and Anja Barth

EU-Reference Laboratory for Pesticide Residues requiring Single Residue Methods, hosted at: CVUA Stuttgart, Schaflandstrasse 3/2, D-70736 Fellbach, Germany; Michelangelo.Anastassiades@cvuas.bwl.de

Ten years following its first publication, QuEChERS has evolved to the most widely used method for pesticide residue control worldwide. Its simplicity, speed, low costs, its amenability to LC and GC applications as well as its versatility have contributed to this popularity. Although QuEChERS provides excellent recoveries of a broad range of spiked pesticides, special attention is necessary as far as the extraction yields of incurred pesticides are concerned. Various factors having an impact on the extraction yields of incurred pesticides were studied such as extraction solvent, temperature, agitation approach, and particle size.

Extraction time and temperature were shown to have the most substantial impact on the extraction yields of incurred pesticides whereas agitation intensity seems to play a less significant role. Extraction yields from samples employed in frozen condition reached a “plateau” after ca. 10-15 min extraction time compared to ca. 2 minutes when employed at room temperature. For practical reasons the use of mechanical shakers is recommended. This minor modification of the QuEChERS method does not significantly alter the manual labor and costs involved.

The extraction yields of incurred pesticides were further shown to strongly depend on their physico-chemical properties. Lipophilic pesticides (high log Kow values) show the most pronounced retardation in their extraction behavior and therefore also the highest yield-increases when extraction times and/or temperature are increased. The impact of prolonging the extraction time from 1 to 15 min, on the extraction yields of incurred pesticides from frozen samples was studied on 132 real crop samples containing 85 different pesticides of a broad polarity range. Out of the 408 pesticide/commodity combinations studied 34% showed yield-increases of more than 25% when increasing extraction times to 15 min (highly significant yield difference). More than half of the 132 tested samples contained at least one pesticide showing a highly significant yield difference between 1 and 15 min extractions. Similar extraction retardation effects were also observed for spiked pesticides but only if these were spiked on commodities with intact surface, not to homogenates thereof.

In addition to QuEChERS, where acetonitrile is employed for extraction, acetone- and ethyl acetate-based methods were also studied. In both cases extraction retardation was noticed with the acetone-based method being less affected. In the case of the ethyl acetate-based method, where extraction is performed in a two-phase system, the highest extraction yield was achieved when employing a high-speed mixer (Ultra-Turrax).

O-9 Assessment of False Positives and False Negatives in the Multiclass, Multiresidue Analysis of Veterinary Drugs in Animal Tissues by UHPLC-MS/MS

Steven J. Lehotay and Alan R. Lightfield

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

Quantitative method validation is well-established to demonstrate accuracy of the method's results, but the often-ignored issue of qualitative validation is probably more important. Consumer and/or environmental safety is at risk when false negatives occur, whereas false positives can cost potentially millions of dollars of unwarranted losses. One way to test for false negatives and positives is through empirical validation just as done quantitatively. In fact, both quantitative and qualitative forms of validation can be done simultaneously. We recently developed a new multiclass, multiresidue method using ultra-high performance liquid chromatography – tandem mass spectrometry (UHPLC-MS/MS) for the analysis of more than 100 veterinary drugs at or below tolerance levels in food animal tissues. This method was validated and approved to meet USDA regulatory criteria for screening and identification purposes, and quantification is a secondary priority because determination of analyte concentrations can be made during confirmation by the official enforcement method. In this study, randomly selected veterinary drugs from the target list of analytes were added at random concentrations near tolerance levels in 50 each of cattle, pork, and chicken muscle tissue extracts obtained with the method. The sample analyses were conducted in blind fashion. Rates of false positives and false negatives were measured depending on pre-defined regulatory LC-MS/MS-based screening and identification criteria. Results showed that the method worked very well for nearly all drugs studied, and the same drugs that gave problems during the previous empirical validation with known spiked samples gave similar difficulties in the blind analyses.

O-10 Implementing Multi-Residue Methodologies for Monitoring Residues in Foods Using LC-MS(/MS)

Simon Hird, Jonathan Guest, Richard Ginn, and George Stubbings

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

The demand for surveillance of a growing number of different food contaminants triggered the need to determine as many analytes as possible using multi-residue methods. Variants of QuEChERS are now commonly used for the determination of hundreds of pesticides in various fresh produce, other agricultural commodities and feed and similar approaches have been applied to cover different contaminants including veterinary medicines and mycotoxins. Such approaches require determination using instrumentation with high selectivity and sensitivity. Although GC-MS(/MS) continues to be used in the analyses of semi-volatiles, developments in LC-MS(/MS) have resulted in powerful instrumentation for determination of the more polar or ionic contaminants. Improved instrument design and LC columns allow more components to be analyzed in the same injection with sufficient sensitivity, within a reasonable short time, using targeted or untargeted analytical approaches. Robust, automated, high throughput, multi-compound analyses is now a reality. There are, however, a number of challenges associated with these approaches. Because the analytes to be monitored are so diverse it is difficult to develop a universal extraction and clean-up method with acceptable recovery for all analytes or to facilitate determination using a single set of analytical conditions. As sample clean-up is minimal or excluded altogether there is an increased chance of failure due to the presence of isobaric interferences and/or ion suppression cause by matrix co-extractives. The rational, benefits and limitations of implementing multi-residue methodologies at Fera are explored in the context of the requirements of different monitoring schemes and operational constraints often encountered.

O-11 Development of an IC-ICP-MS Method to Determine Inorganic Arsenic in Liver from Chickens Treated with Roxarsone

Mary C. Carson[1], Sean D. Conklin[2], Nohora Shockey[3], Kevin Kubachka[3], and Karyn D. Howard[1]

[1]**US FDA, Office of Research, Center for Veterinary Medicine,** 8401 Muirkirk Road, Laurel, MD, 20708, USA; mary.carson@fda.hhs.gov;
[2]US FDA, Office of Regulatory Science, Center for Food Safety and Applied Nutrition,5100 Paint Branch Parkway, College Park, MD, 20740 USA
[3]US FDA Forensic Chemistry Center, Office of Regulatory Affairs, 6751 Steger Drive, Cincinnati, OH, 45237 USA

Roxarsone, (4-hydroxy-3-nitrophenyl)arsonic acid, is an arsenic-containing compound that has been approved as a feed additive for poultry and swine since the 1940s. Work done in the 1960s showed that most tissue residue remains as organic arsenic. However, recent studies using ion chromatography coupled to inductively coupled plasma mass spectrometry showed increased amounts of inorganic arsenic in litter from poultry treated with roxarsone. The FDA wished to re-examine the issue of tissue residues using new technologies. We developed a novel method for the extraction and quantification of arsenic species in chicken liver. A strongly basic

solution solubilized the liver, and ultrafiltration removed macromolecules and particulate material. Ion chromatography separated the arsenic-containing species [arsenite, arsenate, monomethylarsonic acid, dimethylarsinic acid, (4-hydroxy-3-aminophenyl) arsonic acid, (4-hydroxy-3-acetaminophenyl)arsonic acid, and roxarsone] in the extracts, which were then detected by inductively coupled plasma mass spectrometry. The extraction oxidized most arsenite to arsenate. For fortification concentrations at 2 μg kg^{-1} and above, recoveries ranged from 70% to 120%, with RSDs of 7% to 34%. We detected roxarsone, its 3-amino and 3-acetamino metabolites, inorganic arsenic, and additional unknown arsenic species in livers from roxarsone-treated chickens. Both the originating laboratory and a second laboratory validated the method.

O-12 Pesticide Registration Process

This session will include brief presentations made by the following five experts who will discuss various stages of the pesticide registration process: from the initial development of a new pesticide, through various studies that need to be conducted to obtain data necessary for the pesticide registration, including field trials on various crops that are then used to support setting of pesticide US (and other national) tolerances and Codex Alimentarius maximum residue limits. The presentations will be followed by a panel discussion and Q&A session. This is your opportunity to ask the experts and learn more about this complex process!

(1) **New Pesticide Development**
Lesley Czochor
DuPont Crop Protection, Registration and Regulatory Affairs, Stine-Haskell Research Center 300/414, P.O. Box 30, Newark, DE 19714, USA; Lesley.P.Czochor@DuPont.com

(2) **Pesticide Registration Studies**
Thomas Gould
Bayer CropScience, Federal Regulatory Affairs, 2 T.W. Alexander Drive, P.O. Box 12014, Research Triangle Park, NC 27709, USA; tom.gould@bayer.com

(3) **Field Trials and Crop Grouping**
Johannes Corley
IR-4 Headquarters, Rutgers, The State University of New Jersey, 500 College Rd. East # 201 W., Princeton, NJ 08540, USA; jcorley@aesop.rutgers.edu

(4) **Pesticide Tolerance Setting in the USA**
Michael Doherty
U.S. Environmental Protection Agency, Office of Pesticide Programs, Health Effects Division, 1200 Pennsylvania Avenue, N. W., Washington, DC 20460, USA; Doherty.Michael@epa.gov

(5) **Codex MRL Setting and Harmonization**
Yukiko Yamada
Ministry of Agriculture, Forestry and Fisheries, 1-2-1 Kasumigaseki, Chiyoda-ku, Tokyo 100-8950, Japan; yukiko_yamada@nm.maff.go.jp

O-13 Identification of Unexpected Contaminants in Veterinary Practice

Perry Martos and Nick Schrier

University of Guelph, Agriculture and Food Laboratory and Animal Health Laboratory, Laboratory Services Division, 95 Stone Rd. W., Guelph, Ontario, N1H 8J7; Canada; pmartos@uoguelph.ca

Toxic compounds are identified in veterinary practice on a regular basis; exposure to those compounds can lead to physiological responses ranging from no obvious effect to convulsions and to death of the animal. Irrespective of the ultimate physiological response, veterinarians are constantly on the hunt for the cause – was it microbial, natural or chemical in nature. This talk isn't about melamine/cyanuric acid work we carried out in early 2007 to answer the questions then about sick cats. This talk is focused on three case studies involving a dog treated with lipid therapy to treat for a suspected ingested anticoagulant; a cat with a preference for outdoors that presented with convulsions after returning home from her morning prowl; and a dog with strange green eyes and fluorescent pink urine. In all cases, a wide range of physiological observations and analytical techniques were used to identify and confirm the root cause of the animal's health issues.

O-14 Direct MS Detection of Residues, Contaminants and Adulterants in the Food Chain

Hans G.J. Mol, Ruud C.J. van Dam, and Paul Zomer

RIKILT – Institute of Food Safety, Akkermaalsbos 2; 6708 WB Wageningen, Netherlands; Hans.Mol@wur.nl

Liquid chromatography with tandem mass spectrometry is the most widely used technique for the determination of residues and contaminants in the food chain. It is sensitive, selective, and provides quantitative results. In addition, many analytes can be determined in one run which is very effective, especially in the field of pesticide residues. However, there are situations where elimination of the LC from LC-MS is possible and advantageous in terms of cost and speed. The possibilities will be discussed through different types of applications:

1) Determination of highly polar analytes such as certain pesticides (e.g. chlormequat, glyphosate, fosethyl-Al, ethephon, maleic hydrazide). These pesticides require dedicated single residue methods. Including them in the routine scope would substantially increase the capacity demand on LC-MS instrumentation. Furthermore, special LC columns are required which often lack robustness and make chromatography the weakest link of the method. Flow injection–MS eliminates such problems, is much faster (< 1 min) and, with the use of isotopically labeled internal standards, acceptable quantitative results can be obtained.

2) Dietary supplements. Here the use of flow injection combined with full scan high resolution MS is shown to be valuable as rapid screening method for the detection of compounds that may result in harmful effects to the consumer, such as natural toxins or synthetic adulterants.

3) Food forensics. Rapid detection of e.g. illegal pesticides from surface swaps taken at farm sites, or swaps from fruits labeled as organically grown. A comparison with ambient mass spectrometry as alternative option will be made.

O-15 Challenges in Implementing HRMS Screening for Veterinary Drug Residues in a Regulatory Laboratory

Sherri B. Turnipseed,[1] Jack J. Lohne,[1] Wendy C. Andersen,[1] Joseph M. Storey,[2] Susan L. Young,[2] Justin R. Carr,[2] and Mark R. Madson[1,2]

[1]**U.S. Food and Drug Administration, Animal Drugs Research Center**, Denver Federal Center; Denver, CO 80225, USA; sherri.turnipseed@fda.hhs.gov;
[2]U.S. Food and Drug Administration, Denver Laboratory, Denver, CO, USA

High resolution mass spectrometry (HRMS) is a valuable tool for the analysis of chemical contaminants. Our laboratory has developed several methods to screen for veterinary drug residues in food using LC quadrupole time-of-flight (Q-TOF) MS. There have been, however, significant challenges as methods are transferred from the development stage to more routine analysis. Although instrument software programs are designed to evaluate large amounts of information generated by HRMS instruments, it can be difficult to set parameters to detect residue levels of contaminants without generating a large hit list that must be carefully evaluated for false positives. Having experimental retention time and product ion information for analytes facilitates the ability to determine if residues found by the searching software are false detects. This information has been collected for a large number of veterinary drug residues using the Q-TOF MS. Tentative identification of the product ions can be made using a combination of accurate mass, isotope patterns, predictive fragmentation pathways, and the published literature; this data will be useful for the development of veterinary drug residue methods regardless of the MS platform used. Determining how well the compounds of interest are extracted from a matrix with a given analytical method is also important. The screening levels of detection for over 150 veterinary drug residues in milk have been determined, and approximately 60% of those tested can be detected at concentrations of 10 ng/mL or less; over 70% can be found when fortified into milk at 100 ng/mL.

O-16 Triumphs and Challenges of High Resolution Mass Spectrometry in Comprehensive Pesticide Residue Screens

Mark R. Crosswhite, Ghislain Gerard, and Walter Hammack

Florida Department of Agriculture and Consumer Services, 3125 Conner Boulevard, Lab 3; Tallahassee FL 32399 USA; mark.crosswhite@freshfromflorida.com

Triple quadrupole mass spectrometers are used extensively in pesticide residue analysis because of their capabilities of providing a high degree of confidence in identification and quantification of analytes, particularly because of MS^n experiments. We find that a well optimized triple quad can be used to analyze roughly 150 compounds over a 12 minute liquid chromatographic run. However, the duty cycle limitations of such instruments become increasingly difficult to manage as the number of pesticides in our screen increases.

As our laboratory is continually expanding our analytical capabilities we can no longer solely rely on triple quadrupole instruments. In our efforts to approximately double the number of pesticides in our screen we incorporated an orbitrap mass spectrometry, which is a fundamentally different analytical platform in that it has mass resolving power of ~100,000 and continually scans over a large mass-to-charge range, but cannot perform MS^n experiments. We find that the orbitrap has the potential for transformational improvements in our analytical capabilities. Highlighted here are the areas where we believe this platform will excel, problems which we have been able to resolve and issues that require complementary techniques.

O-17 Florida's 2013 Chemical Residue Program in Review

Jo Marie Cook

Florida Department of Agriculture and Consumer Services, 3125 Conner Blvd., #3, Tallahassee, FL 32399, USA; jomarie.cook@freshfromflorida.com

Florida's Chemical Residue Laboratories conduct testing for pesticides in fresh fruits and vegetables and enforce federal pesticide tolerance regulations of 40 CFR 180. For the last two years they have also screened seafood for polycyclic aromatic hydrocarbons (PAHs) and the dispersant dioctylsulfosuccinate (DOSS) in response to the oil spill. In addition, they are implementing a custom designed LIMS system. A summary of pesticide residue analyses and tolerance violation case studies will be presented as well as a summary of seafood screening. Plans for the future will include a discussion of the legal implications of screening for antibiotics in honey and seafood.

O-18 Pesticide Residues in Produce: The Connecticut Market Basket Survey

Brian D. Eitzer,[1] Walter J. Krol,[1] Terri Arsenault,[1] Jason C. White,[1] and Ellen Sloan[2]

[1]**The Connecticut Agricultural Experiment Station**, 123 Huntington St., New Haven, CT 06511, USA; brian.eitzer@ct.gov,
[2]The Connecticut Department of Consumer Protection, Hartford, CT, USA

The Connecticut Agricultural Experiment Station, in conjunction with the Connecticut Department of Consumer Protection (DCP), has been conducting a market basket survey of pesticide residues in produce for over 30 years. Each year inspectors from the DCP bring in over 200 samples of a diverse range of produce including: fresh and frozen produce; foreign and domestic (including locally grown) produce; conventional and organic produce. These products are all analyzed for a wide variety of pesticides. The analytical methods used have changed over the years and currently consist of a QuEChERS extraction with both GC/MS and LC/MS detection. Method improvements have led to both a higher proportion of the positive findings and a higher proportion of violative samples. Historical and recent results will be discussed along with a discussion of recent changes to analytical procedures to incorporate both LC-high resolution mass spectrometry, and GC/MS-MS.

O-19 Montana's Program for Monitoring Trace Level Pesticides in Groundwater

Jona Verreth, Angela Schaner, and Heidi Hickes

Montana Department of Agriculture, MSU, McCall Hall, Bozeman, MT 59717, USA; jverreth@mt.gov

For over 20 years the Montana Department of Agriculture (MDA) has been monitoring trace level pesticides in groundwater in compliance with the State of Montana Groundwater Protection Act of 1989. A total of 42 permanent monitoring wells and a number of special projects have generated data for the program. In 2006, in an effort to provide more meaningful information, the MDA Analytical Laboratory developed and implemented a Universal Method to capture as many compounds as possible with a broader range of acceptable performance. The method includes a SPE clean-up and concentration step and utilizes UPLC-ES/MS/MS to analyze for approximately 100 pesticides. A presentation of method improvements made over the years, results generated and application of those results will be discussed.

O-20 2012 Indiana's Bee Kill Investigations: Field Observations, Sampling and Pesticide Residue Analysis

Ping Wan

Office of Indiana State Chemist, 175 S. University Street; West Lafayette, IN 47907, USA; wanp@purdue.edu

During the spring season of April to May 2012, seven unrelated bee kill incidents were reported to the Office of Indiana State Chemist (OISC). All incidents reported large quantities of dying/dead bees. Complainants (beekeepers) suspected that the bee deaths were in

response to either pesticide application or corn planting in neighboring fields. Dead bees and in some cases, bee pollen and vegetation from the target planting fields were sampled and tested for pesticide residue. All samples were detected with an insecticide of the neonicotinoid class named clothianidin, in the levels from 2-80 ppb.

The impact of neonicotinoid pesticides on bee health remains controversial. Research reports released by the European Food Safety Authority have linked neonicotinoid pesticides, in particular clothianidin, with poor bee health. However communications released by Bayer CropScience pointed out that there is an increasing consensus in scientific research that poor bee health and colony losses are caused by multiple factors, the parasitic Varroa mite being the key issue.

O-21 Two current topics of food and feed contamination: Identification and Quantification of 6 Illegal Antibiotics in Chinese Chicken Jerky Dog Treats and the Identification of Illegal Colors in Various Food Products

Robert Sheridan, Jenifer Kemnah, Kristen Hafler, and Thomas Tarantelli

New York State Department of Agriculture and Markets, 6 Harriman Campus Road; Albany, NY 12206, USA; robert.sheridan@agriculture.ny.gov

Since 2007 Chicken jerky dog treats imported from China have been implicated in causing illness and death in dogs in several countries. Using LC/MS/MS targeted screens and LC/TOF analysis six un-allowed antibiotics have been identified and quantified in several brands of chicken jerky treats imported from China to the US. Each bag of treats was sub-sampled so that each individual treat was considered a unique sample which was ground and subjected to extraction and analysis. This was done to aid in the identification of potential hot spots of contamination since a bag of 60 treats may be from unrelated sources. Trimethoprim, tilmicosin, enrofloxacin, sulfaquinoxaline and sulfamethoxazole were detected using a targeted screen and sSulfaclozine was detected using a combination of a targeted screen and high resolution LC/MS. While the six antibiotics are most likely not responsible for the dog illnesses the products have been removed for sale in the US since they were in violation of FDA tolerances.

As the world's food supply becomes increasingly globalized much of the food now available to consumers in the US originate from countries with a poor history of food safety. One of the most common adulterants in food remains the use of illegal colors in food because many are much cheaper than the legal alternatives. The identification of illegal colors in food has traditionally been accomplished with thin layer and paper chromatography however many industrial solvent dyes cannot be detected in this way. We have developed a UPLC/MS/MS method which can detect 29 un-allowed solvent and industrial dyes in various foods to use as an addition to our paper and thin layer color methods. Using this method we have identified Rhodamine B, Auramine O, Metanil Yellow, Sudan I, Orange II, Malachite Green, Brilliant Green, Crystal Violet and Basic Blue 3 in various imported food products.

O-22 Moving Food Safety to Peptides- New Approaches using LC-MS for the Detection of Plant Allergens

Robert D. Voyksner[1], Jerry Zweigenbaum[2], and Jennifer A. Sealey-Voyksner[1]

[1]**LCMS Limited,** 1502 West Hwy NC- 54, Suite 504, Durham, NC 27707, USA; robert_voyksner@lcmslimited.com
[2] Agilent Technologies Inc., 2850 Centerville Road, Wilmington, DE 19808, USA

Plant-based allergens make up half of the FDA's list of priority allergens. Wheat, soy, peanut and tree nuts are food allergens affecting greater than 4% of the North American population. The food industry currently relies on various ELISA methodologies to detect the various food allergens, but these methods only target single analytes and can suffer drawbacks, such as lack of sensitivity and negative/positive bias in their quantitative results. Recently, LC-MS methodologies have been developed utilizing tryptic digests for the detection of some plant-based allergens, based on the detection of representative marker peptides. To date, there has been virtually no work published on the characterization of allergenic plant proteins using other enzymes to digest the proteins for marker peptide discovery. This presentation will discuss the development of an analytical method capable of the quantitative detection trace levels of peptide markers representing 13 plant allergens. This work utilized pepsin, chymotrypsin and trypsin to digest the plant proteins. Marker peptides were produced with better sensitivity and were less affected by food processing steps. LC-QTOF accurate mass and MS/MS analysis permitted the identification of the marker peptides from the digested plant proteins. This method was tested on a wide variety of foods products to validate the specificity of each peptide marker and whether it was detected in both cooked and uncooked foods with no false positives or negatives. This LC-MS method allows accurate and sensitive detection of multiple allergens in a single assay at concentrations down to ppb ranges.

O-23 Environmental Analysis and the Exposome Phase I: Chemical Cartography using GC/Q-TOF Mass Spectrometry

Anthony Macherone[1,2]

[1]**Agilent Technologies, Inc.**, 2850 Centerville Road; Wilmington, DE 19808, USA; anthony_macherone@agilent.com;
[2]**The Johns Hopkins School of Medicine**, 725 N. Wolfe Street; Baltimore, MD 21205, USA; amacher1@jhu.edu

The exposome has been defined as the sum of all exogenous exposures to food, air, water, lifestyle choices *and* endogenous exposures to xenobiotics, biosynthesized chemicals and metabolites over a complete lifetime. Exposomics is the global measurement of the exposome using "omics" tools to correlate environmental exposure to disease. The inherent complexity of examining the environment for contaminates like total persistent organic pollutants (POPs) and correlating exposure to human health will require an interdisciplinary approach including but not limited to genomics, metabolomics, epidemiology, toxicology, clinical chemistry, food safety monitoring and analytical environmental chemistry. The instrumentation required to examine these samples are just as diverse and include liquid chromatography, gas chromatography, mass spectrometry, inductively-coupled plasma mass spectrometry and nuclear magnetic resonance spectroscopy and other spectroscopic techniques. Using the exposomics model, a proof of principle study is described for the creation of global discovery gas chromatography- time of flight and quadrupole time of flight mass spectrometry (GC/TOF & GC/Q-TOF) methods to identify, measure and confirm the breadth of POPs, endocrine disruptors and other chemical classes in multiple locations of a specified region and geographically mapping the resulting data. The chemical cartographic data will be correlated to public health information to develop an exposure potential. Public health information may then be correlated to begin to associate disease with exposure e.g., the genome:exposome relationship. The development of an interactive map that illustrates the chemical composiiton of an enviroment and correlation to disease will aid in the understanding of how environmental exposure influences public health.

O-24 Can Oyster Harvest Areas Be Identified By Their Cadmium- Lead Signature?

Marc E. Engel

Florida Dept. of Agriculture and Consumer Services, 3125 Connor Blvd #9, Tallahassee, FL 32399, USA; Marc.Engel@freshfromflorida.com

The American oyster, *Crassostreavirginica*, is an economically and environmentally important estuarine foundation species. As sessile filter feeders, they are exposed to a variety of anthropogenic contaminates characteristic to their location. Over 400 oysters have been analyzed for cadmium and lead since 2011. A cadmium and lead signature (Cd-Pb_Sig) can be assigned to their harvest area from the following metrics when oyster tissue are analyzed: average cadmium (Cd) concentration, average lead (Pb) concentration and average Pb concentration/average Cd concentration (Pb/Cd). This method shows promise as a screening tool for identifying oyster harvest location on a broad scale (i.e. between states, estuarine areas) for environmental monitoring, regulatory and law enforcement purposes. These screening results will have to be confirmed by a high resolution isotope ratio mass spectrometry.

Whole, live oysters were collected by Florida Department of Agriculture and Consumer Services (FDACS) inspectors from wholesale and retail outlets typically within days of harvest. Oysters were harvested from the coastal waters of Florida (FL), Louisiana (LA), Texas (TX), and Virginia (VA). Samples were shipped overnight to FDACS Food Safety Laboratories and frozen until analysis. Harvest location, shell length, and flesh weight were recorded for all samples prior to homogenization. All samples were analyzed in duplicate and were closed vessel microwave digested with HNO_3. Gold and internal standards (Rh, Lu) were added to all samples and standards. For each set of 10 samples; a reagent blank, calibration check standard and SRM 2976 Mussel Tissue (NIST) were analyzed. All samples were analyzed by ICP-MS.

O-25 Moving Forward Together To Enhance Global Product Safety: A Laboratory Perspective

Sarah E. McMullen [1,2] and Carl Sciacchitano[1]

[1]**US Food and Drug Administration,** Office of International Programs, 10903 New Hampshire Avenue Building 31/32, Silver Spring, MD 20993, USA; sarah.mcmullen@fda.hhs.gov;
[2]US Food and Drug Administration**,** Southeast Regional Laboratory, 60 Eighth St. NE; Atlanta, GA 30309, USA

In 2011, FDA released a special report entitled "Pathway to Global Product Safety and Quality" highlighting the need to partner with nations globally to enhance global product safety/quality. In addition, FDA recently released its International Food Safety Capacity Building Plan which addresses both the acceptance of laboratory methods across the international community and the exchange of

information on current and new methods. As we move forward together to enhance product safety, we see a great number of opportunities to leverage existing best practices, share communication/informational platforms, and establish new/novel approaches for future collaborations.

O-26 Understanding the Food Safety Modernization Act in Relation to Chemical Risks

David W. K. Acheson

Leavitt Partners LLC, 299 South Main Street, Salt Lake City, UT 84111, USA; david@leavittpartners.com

The Food Safety Modernization Act was signed into law in January 2011 and represents the most significant change to the regulation of FDA regulated food products in over 75 years. The primary focus of the new legislation is to focus the emphasis for food safety on prevention. The impact is wide but is principally focused on growers, manufacturers and processors putting food into interstate commerce in the United States whether they be domestic or foreign firms. In January 2013 the FDA released the proposed preventive control rules which will impact many FDA registered firms. These new rules will require food manufactures and processors to develop a food safety plan that not only identifies risks but documents how these risks are controlled. The risks to be assessed include, microbiological, chemical, physical and radiological. The FDA expects food companies to understand these risks and establish effective preventive controls that are validated. As part of this process food companies will have to maintain detailed records of both the assessment and the on-going monitoring. Globally these rules will impact food manufacturers all over the world if they are supplying food into US commerce. FSMA is complex and far reaching and while an important area to understand and comply with should not be taken in the absence of the broader need for a focus on risks that can impact a company's brand.

POSTER ABSTRACTS

P1 What Pesticides Have You Eaten Today?

Liesl Krone[1] and André Schreiber[2]
[1]**Action Middle School**, 1300 James Rd, Granbury, TX; jennifer.krone@absciex.com
[2]AB SCIEX, Concord, ON, Canada

There is a lot of concern about ingesting pesticides that could be left on fruits and vegetables. Pesticides are designed to be toxic, and their effects on people aren't very well understood, so it's best to avoid them when you can.

What fruit contains the most pesticides and does peeling them or washing them truly make your fruits pesticide free before you eat them?

In summary, in this study we found out that organic is not truly organic. The pesticide levels were low and way below the maximum tolerance but they were still present. Also, washing your grapes just with water does not make much of a difference. Maybe they need to be washed with something stronger than water, like soap. The peel of the oranges and bananas contained most of the pesticides so it is good that we don't eat the skins! Some recipes ask for orange and lemon zest, this most likely adds more pesticide contamination into your food. It was also found that the outer skin does not block all the absorption of the pesticides since chemicals were found in the pulp. The mass spectrometer was able to detect very low levels of chemicals, even those that had been banned or used outside the US. Be careful of imported fruits, they may contain pesticides that are not approved in the US.
It is still better to eat fruit than junk food but maybe we need to eat fewer grapes! Grapes, especially imported ones, are listed as part of the Dirty Dozen. In our study Grapes were the dirtiest!

P2 Cell Culture Studies of Exposure Response: Highlighting Biochemical Pathway Changes

Joel Gummer[1,2], Catherine Rawlinson[1,2], Bruce Peebles[1,2], Katherine Roussety[1,2], Garth Maker[1,2,3], Ian Mullaney[1,3], and Robert Trengove[1,3]
[1]**Separation Science & Metabolomics Laboratory, Murdoch University**, 90 South Street, Murdoch, WA 6150, Australia; r.trengove@murdoch.edu.au;
[2]Metabolomics Australia, Murdoch University Node, Murdoch University, 90 South Street, Murdoch, WA 6150, Australia;
[3]School of Veterinary and Life Sciences, Murdoch University, 90 South Street, Murdoch, WA 6150, Australia

Animal and human exposure to residues and POPs can result in measurable changes in metabolic profiles and lipid profiles. For animals, animal models, and humans there are "biomarkers" of exposure, however interpreting the impacts at the molecular level for whole animal and human studies is challenging and typically limited to relatively few "biomarkers". Cell culture studies using immortalized cell lines can be used to dissect the changes in biochemical pathways following exposure to individual and combined chemical challenges. In addition, mechanisms of protective response and evaluation of "protective treatments" can be studied. Cell lines are available for a wide range of animal and human organs and these can be used in conjunction with biofluids to target exposure impacts at the organ/sub organ level. In addition, the immortalized cells can be characterized to identify receptor targets to further elucidate repose and protective mechanisms. The changes observed in immortalized cell lines can then be validated using isolated cells.

The combination of untargeted (semi-quantitative) and targeted approaches can be used to identify key biochemical pathways and pathway changes resulting from exposure. Immortalized cell lines are used to study exposure to individual chemicals and "cocktails" of chemicals and the link to potential health impacts. Tools developed to highlight and monitor pathways using triple quadrupole mass spectrometers will be presented.

P3 Development and Validation of a Multi-Residue Method for the Determination of 340 Pesticides in Agricultural Products using the GC-MS/MS Triple Quadrupole Technique

André de Kok and Barbara Kiedrowska
NVWA - Netherlands Food and Consumer Product Safety Authority, Laboratory of Food and Feed Safety - Chemistry Laboratory, National Reference Laboratory (NRL) for Pesticide Residues in Food and Feed,
Wageningen, The Netherlands; andre.de.kok@vwa.nl; barbara.kiedrowska@vwa.nl

Continuous development of gas chromatography (GC) coupled to triple quadrupole mass spectrometry (GC-MS/MS TQ) operated in multiple reaction monitoring (MRM) mode has resulted in a trend towards pesticide analysis methods for an ever increasing number of compounds within a single injection and chromatographic run. An analytical method combining the very fast and efficient Dutch Mini-Luke extraction technique and direct GC-MS/MS TQ detection, without any cleanup, was developed for the determination of 340 pesticides (including some metabolites/degradation products and internal standards) in fruits and vegetables. A homogeneous 15 g sample is extracted with 30 mL acetone (30 sec via a Polytron homogenizer), followed by a partitioning step (again 30 sec via

a Polytron homogenizer) with 60 mL dichloromethane / petroleum ether (1:1) and Na_2SO_4, using the salting-out effect in order to assure good recoveries for polar pesticides. The mixture is centrifuged (5 min at 3500 rpm), an aliquot (6 mL) of the extract is then evaporated to dryness (batchwise, on a water bath) and the residue is redissolved in 1 mL isooctane/toluene (9:1) and 5 µL is injected (via LVI-PTV) into the GC-MS/MS TQ. The method was developed on both a Varian 320 GC-MS/MS TQ instrument and the newest generation Bruker Scion GC-MS/MS instrument, using 680 transitions during a 35-min run time. Various GC- and MS-parameters were tested and optimized in order to obtain the highest sensitivity.

Initial validation of the method was carried out for 43 representative pesticides and two representative matrices (lettuce and orange). Recovery studies were performed at spiking levels of 0.01, 0.02 and 0.05 mg/kg. All analytes, except those that tend to degrade in the GC injector (captan and folpet), met the EU DG SANCO method validation criteria (i.e. average recoveries in the range 70-120%, with RSD <20%), even at the lowest tested spike level of 0.01 mg/kg. Thus, validated LOQs of 0.01 mg/kg were easily achieved. Calibration curves were linear over the range 0.5-100 ng/mL, with $r^2 > 0.98$ for all compounds, except for chlorothalonil in the matrix orange. The estimated instrument limit of detection for the majority of analytes was well below 1 ng/mL for both matrices. We did not observe any significant interferences in the MS/MS traces, which proves the high selectivity of the validated method. The method has been applied successfully in routine analysis of fruits, vegetables, cereals and various types of other difficult matrices.

P4 The Art of Method Development: Modification of an Analytical Method for the Analysis of a Clopyralid in Animal Tissues and the Pitfalls Encountered.

Stanley R. Shaffer[1], Mark Fortenberry[1], Brian Bothwell[1], Darcy Shackelford[2] and Ed Olberding[3]
[1]**Analytical Bio-Chemistry Laboratories,** 7200 East ABC Lane; Columbia, MO 65202, USA; shaffers@abclabs.com
[2]Dow AgroSciences, Inc.; [3]Haystack Analytics, LLC

The objective of the study was to convert a method utilizing analyte derivatization with GC/MS detection to a direct analysis by LC/MSMS. The analytical method consisting of a basic hydrolysis/extraction technique followed by HLB solid-phase extraction (SPE) column (Waters, 200 mg/6 mL) cleanup, propylation, and detection by capillary gas chromatography with negative-ion chemical ionization mass spectrometry (GC-NCI-MS) was modified for direct analysis by LC/MSMS. The original extraction procedure and a modification of the HLB SPE cleanup procedure were utilized to provide successful analysis by LC/MSMS without matrix enhancement/suppression or interferences. Conversion of a method from one utilizing derivatization followed by GC/MS analysis to that of a direct detection by LC/MSMS can pose unique analytical challenges to obtain comparable sensitivity and specificity. This presentation will give an overview of the unique recovery, interference and sensitivity issues overcome during the process.

P5 Pesticide and Veterinary Drug Residue Analysis by Multiple Walled Carbon Nanotubes (MWCNTs) and multi-Plug-Filtration-Cleanup (m-PFC) Method

Canping Pan[1], Pengyue Zhao[1], Yanjie Li[1], Aijuan Bai[1], Di Guan[1], Yahui He[1] and Xuesheng Li[2]
[1]**Department of Applied Chemistry, China Agricultural University**, Beijing 100193, P R China; panc@cau.edu.cn;
[2]Department of Agriculture Science, Guangxi University, P R China

Pesticide and veterinary drug residue analysis is advancing very rapidly, which is required by the monitoring market of various countries in domestic and international trading, risk assessment of dietary intakes, or environmental research etc. The key procedure of this is the cleanup of sample extracts in order to avoid interferences from complex matrices. In our study, multiple walled carbon nanotubes (MWCNTs) were introduced into the cleanup methods for pesticide residues, especially for difficult samples. Thus, several types of MWCNTs were evaluated for their potential of absorbing pesticide compounds or matrix interferences. Function-modified MWCNTs were selected and used as absorbents in reverse dispersive cleanup methods for acetonitrile, methanol, water, ethyl acetate etc. extracts of vegetables, fruits, teas, juice, milk, and meat. Several types of pesticides and veterinary drugs were examined in these samples during the method development and validation. It was found that MWCNTs or these materials mixed with C18, PSA, NH2 or GCB could be used as powerful absorbents for cleanup procedure with better results than using PSA, C18 and some other materials alone. Furthermore, the authors developed new multi-plug-filtration cleanup methods (m-PFC) for rapid application. This method has a high potential to be widely applied for monitoring of pesticides at trace levels in the future for various agricultural commodities.

P6 Laser Diode Thermal Desorption Mass Spectrometry for Veterinary Drug Residue Analysis

Wendy C. Andersen,[1] Jack J. Lohne,[1] Sherri B. Turnipseed,[1] Susan B. Clark,[2] Joe M. Storey,[1] and Mark R. Madson[1,2]
[1]**Animal Drugs Research Center** and [2]Denver Laboratory, U.S. Food and Drug Administration, W. 6th Ave and Kipling St., DFC Bldg 20, Denver, CO, 80225-0087, USA; wendy.andersen@fda.hhs.gov

Laser diode thermal desorption atmospheric pressure chemical ionization mass spectrometry (LDTD-APCI-MS/MS) is a direct sample introduction MS analysis technique in which an infrared laser is used to thermally desorb analytes from sample extracts dried onto metal surfaces of a 96-well Lazwell plate. Gas phase analytes are then ionized by APCI and analyzed by MS/MS. Without chromatographic separation, the per sample analysis time is on the order of seconds. Quinolone antibiotics (flumequine, oxolinic acid and nalidixic acid) were quantitatively determined and qualitatively identified in fish extracts using LDTD-MS/MS. The technique provided linear (R^2>0.99) calibration curves from 0 to 100 ng/g, recoveries above 80%, and precision less than 20% RSD. The per

sample analysis time was less than 14 seconds per sample and detection levels for these antibiotic residues in catfish, shrimp, and salmon ranged from 1.5 to 7 ng/g. LDTD-MS/MS has also been used for the analysis of sulfonamides in milk and chloramphenicol in honey. Application of LDTD-MS/MS to other compound classes may provide rapid veterinary drug residue screening in foods and feeds.

P7 Analysis of Multiclass Veterinary Drug Residues in Baby Food by Ultra Fast Chromotagraphy with High Performance Triple Quadropole Mass Spectormetry

Charles Yang, Dipankar Ghosh, and Mary Blackburn
Thermo Fisher Scientific, 355 River Oaks Parkway, San Jose, CA; charles.yang@thermofisher.com

The quantification of multi-class veterinary drug residues (sulfadiazine, levamisole, marbofloxacin, trimethoprim, sulfadimidine, enrofloxacin, tetracycline, thiabendazole, danoflozacin, oxytetracycline, sulfachloropyridazine, diflozacin, sarafloxacin, sulfadimethoxine, chlorotetracycline, oxolinic acid, sulfaquinoxaline, tilmicosin, oxfendazole, doxycycline, flumequine, tylosin, mebendazole, erythromycin, josamycin, albendazole, fenbendazole, emamectin, and ivermectin) from baby food 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 advantages to this approach with very little sample cleanup necessary prior to injection, as well as a short run time on the LC/MS and to have a robust method to meet new regulation requirements. 10 uL injections of extracted baby food containing many veterinary drugs were injected onto C18 column. Compounds of interest were eluted using a ballistic gradient elution profile. A high performance triple quadrupole mass spectrometer with a heated electrospray source (HESI), was used to analyze compounds of interest in both positive and negative ionization, and the data are collected, analyzed, and reported using a customized software. To test the assay, standard curves with seven points were prepared in different baby foods covering the range 10 pg/mL (ppt) to1 µg/mL (ppm). Two ions were monitored, one for Quantitation and the other for Qualification. The calibration curves were linear over the ranges described above. The columns showed no deterioration in quality or performance when using the multiple baby food matrices.

P8 Analysis of Targeted and Non-Targeted Identified Contaminants in Storm Water Retention Ponds using LC-HRMS with Online Solid Phase Extraction

Gordon Getzinger,[1] P. Lee Ferguson,[1,] Jonathan Beck,[2] Charles Yang,[2] Frans Schousten[2], and Jennifer Massi[2]
[1]Duke University; Durham, NC, USA; [2]**Thermo Fisher Scientific**; 355 River Oaks Parkway, San Jose, CA, USA;
jennifer.massi@thermofisher.com

We have examined the occurrence and distribution of wastewater-derived and turf grass management organic contaminants in storm water retention ponds impacted by sewage-derived irrigation water and located on a coastal golf course community at Kiawah Island, SC. Targeted LC-HRMS methods were developed for ~30 organic micropollutants including known xenoestrogens, pesticides, herbicides, and fungicides. Furthermore, a LC-HRMS/MS non-targeted screening workflow, utilizing a comprehensive, accurate mass MS/MS spectral library of ~1000 unique substances relevant to environmental systems, was implemented to monitor the occurrence of micropollutants for which compound specific instrumental parameters had not been developed. The quantitative methodology utilized an online solid-phase extraction approach with two HPLC pumps and two HPLC columns, one for sample pre-concentration and the other for analytical separation of analytes prior to introduction into a triple quadrupole mass spectrometer. This online sample preparation configuration eliminated the need for conventional offline solid-phase extraction; increasing throughput and reducing error incurred through offline sample preparation. This poster will focus on the quantitative aspect of the project.

P9 Identification of illegal colors and dyes in various food products by LC/MS/MS

Robert Sheridan and Thomas Tarantelli
New York State Department of Agriculture and Markets, 6 Harriman Campus Road; Albany, NY 12206, USA;
robert.sheridan@agriculture.ny.gov

The world's food supply is becoming increasingly globalized and much originates from countries with a poor history of food safety. One of the most common adulterants in food remains the use of illegal colors because many are much cheaper than the legal alternatives. The identification of illegal colors in food has traditionally been accomplished with thin layer and paper chromatography. Colors such as Yellow 6, yellow 5, Red 40, and blue 1 and 2 can be determined by these techniques and with the aid of ultraviolet long and short wavelength reflection. Other families of food colors such as solvent dyes, reactive dyes and fluorescent brighteners cannot be determined in this way and require additional detection methodology. We have developed a UPLC/MS/MS method which can detect 29 un-allowed solvent and industrial dyes in various foods to use as an addition to our paper and thin layer color methods. The UPLC/MS/MS method monitors two precursor/product transitions per compound for ion ratio confirmation after a simple liquid extraction. Using this method we have identified Rhodamine B, Auramine O, Metanil Yellow, Sudan I, Orange II, Malachite Green, Brilliant Green, Crystal Violet and Basic Blue 3 in various imported food products. None of these compounds are approved for use in food and some are very toxic such as Auramine O which is not only a dye but in some countries is used as a means of committing suicide.

P10 Identification and quantification of 6 illegal antibiotics in Chinese chicken Jerky dog treats

Robert Sheridan, Jenifer Kemnah, and Kristen Hafler
New York State Department of Agriculture and Markets, 6 Harriman Campus Road; Albany, NY 12206, USA; robert.sheridan@agriculture.ny.gov

In 2007 Chicken jerky dog treats imported from China were first implicated in causing illness and death in dogs in several countries. Chicken jerky treats are typically sold as bags of between 10 and 60 individual pieces. Common practice with samples of this type is to grind several pieces and homogenize the resulting ground portion as one sample. When these samples were subjected to targeted screens occasional detections of sulfaquinoxaline were seen at relatively low levels and inconsistently. We then began treating each individual jerky treat as a unique sample. This was done to aid in the identification of potential hot spots of contamination since a bag of 60 treats may be from unrelated sources. The samples were extracted with acetonitrile, partitioned with water and salt, concentrated and filtered before LC/MS/MS analysis. Identification and quantitation of sulfaquinoxaline, sulfamethoxazole, enrofloxacin, trimethoprim and tilmicosin was accomplished using targeted screens monitoring 2 precursor product transitions per analyte. Sulfaclozine was identified with the assistance of LC/TOF accurate mass measurement which aided in the generation of an empirical formula. During targeted antibiotic screening a signal was seen in some samples for both the target and qualifier ions of sulfachloropyridazine however the ion ratio and retention time was a poor match. LC/TOF investigation of this unidentified compound concluded this peak had the same empirical formula as sulfachloropyridazine. One of the isomers of sulfachloropyridazine is another sulfonamide antibiotic, sulfaclozine. After purchasing this standard it was confirmed as the unknown compound and added to our targeted screen of 6 consistently detected antibiotics. Two precursor product transitions were monitored for each analyte and quantitation was performed using matrix matched external standards. Nearly all bags of treats from China contained at least one of the six un-allowed antibiotics in subsamples at concentrations as high as 1500 ng/g. Five chicken jerky products that were manufactured in the US were investigated by sub-sampling 10 jerky treats from each bag and analyzing each treat. All 50 samples were free of all six analytes. While the six antibiotics are most likely not responsible for the dog illnesses the products have been removed for sale in the US since they were in violation of FDA tolerances.

P11 Identification Criteria for Residues Determined by LC-MS/MS: Are They Fit-for-Purpose?

Hans Mol[1], Paul Zomer[1], Monica Garcia Lopez[2], Richard Fussell[2], Jos Scholten[3], Andre de Kok[3], Anne Wolheim[4], Michelangelo Anastassiades[4], Ana Lozano[5], and Amadeo Fernandez Alba[5]
[1]**RIKILT – Institute of Food Safety**, Wageningen, Netherlands; hans.mol@wur.nl
[2] Food and Environmental Research Agency, York, UK
[3] NVWA – Netherlands Food and Consumer Product Safety Authority, Wageningen, Netherlands
[4] CVUA, Stuttgart, Germany
[5] University of Almeria, Almeria, Spain

LC-MS/MS is one of the most widely used techniques for identification (and quantification) of residues. Although the same technique is applied, the parameters and criteria for identification vary depending on where in the world the analysis is performed and/or the purpose (e.g. pesticides, veterinary drugs, forensic toxicology, sports doping). For pesticides in food/feed analyzed by LC-MS/MS in the EU, according to SANCO/12495/2011, identification is based on relative retention time and the ion ratio of ≥2 transitions, which need to fulfill certain criteria. These originate from EU Commission Decision 657/2002 which in turn were was in essence based on expert opinions. Since then, sample preparation became more generic, and LC-MS/MS matured and became a routine technique. However, the criteria remained the same. In the frame of the biannual revision of the SANCO guidance document, the deviations of retention time and ion ratios relative to reference values based on solvent standards have been systematically assessed for pesticides in fruits and vegetables. The study involved ~120 pesticides (two transitions each) varying widely in polarity, sensitivity, and m/z of the transitions; 20 different matrices; blanks and spikes at 0.01, 0.05 and 0.20 mg/kg. The sample extracts and solvent standards were analyzed as one 120-injection sequence in five laboratories using different LC-MS/MS systems. The total data set consisted of responses observed in over 130,000 extracted ion chromatograms. The results clearly indicate that the current identification parameters and criteria are obsolete and need to be revised, and provide scientifically based input for new criteria.

P12 Validation of a Qualitative Screening Method for Pesticides in Fruits and Vegetables by GC-(APCI)QTOF-MS

Tania Portoles[1,2], Hans G. J. Mol[2], J.V. Sancho[1], and Félix Hernández[1]
[1]Research Institute for Pesticides and Waters, University Jaume I, Castellón, Spain; tportole@uji.es
[2]**RIKILT – Wageningen UR**, Institute of Food Safety, Wageningen, Netherlands; hans.mol@wur.nl

GC with full scan MS is a powerful approach for wide-scope screening of pesticides and contaminants. Nominal resolution MS has been used for this purpose for a long time. High resolution/accurate mass TOF is a more recent development offering a higher selectivity. Dedicated GC-MS systems involve electron ionization (EI) typically resulting in a strong fragmentation. This prohibits a straightforward detection of analytes in the raw data through their (quasi)molecular ion and is a serious drawback in detection of unknowns. Recently, atmospheric pressure chemical ionization (APCI) has become available as an alternative option to couple GC to MS. This results in much less fragmentation compared to EI, is more generic than PCI/NCI, and provides access to a range of existing MS systems initially developed for LC-MS. In this work we have applied GC-(APCI)QTOF-MS instrumentation for screening and identification of pesticides in fruits and vegetables. Non-target acquisition was performed by alternating scan events: one at low

collision energy and another at a collision energy ramp (MS^E). This way both (quasi)molecular ions and fragment ions were obtained. The qualitative screening method was validated according to SANCO/12495/2011. To this end 20 samples (10 samples in duplicate) were spiked at 10, 50 and 200 µg/kg with 150 pesticides and analyzed. Samples selected were tomato, lettuce, cucumber, pepper, oranges, grapes, peach, apples, cauliflower and carrots. The screening detection limit (SDL) of the method was established for each of the pesticides. In addition, the possibilities for identification according to the SANCO guideline were assessed.

P13 A Miniaturized Residue Analytical Method for the Determination of Zoxamide and its Two Acid Metabolites in Ginseng Using LC-MS/MS

Lynda Podhorniak
U.S. EPA Analytical Chemistry Laboratory; 701 Mapes Road; Fort Meade, MD 20755, USA; podhorniak.lynda@epa.gov

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.

P14 Multi-Residue Method for the Analysis of >140 Pesticide Residues in Fish using Fast, Low-Pressure GC-MS/MS

Yelena Sapozhnikova
USDA Agricultural Research Service, Eastern Regional Research Center, 600 East Mermaid Lane, Wyndmoor, PA, 19038, USA; yelena.sapozhnikova@ars.usda.gov

A multi-residue method for the analysis of over 140 pesticide residues in fish was developed and evaluated using fast, low pressure gas chromatography triple quadrupole tandem mass spectrometry (LP-GC/MS-MS). The method was based on a QuEChERS (quick, easy, cheap, effective, rugged, safe) extraction with acetonitrile and dispersive solid-phase extraction (d-SPE) clean-up with zirconium-based sorbent Z-sep from Supelco. Sample preparation for a batch of 10 homogenized samples takes less than 2 hours per analyst. Fast LP-GC/MS-MS allows for a chromatographic run of 9 min achieving high throughput. Fenthion-d_6 was used as an internal standard for quantification of pesticides. The detection limits were 0.5-5 ng/g. The developed method was evaluated at four spiking levels (1, 5, 50 and 100 ng/g) for five replicates per level. Acceptable recoveries (70-120%) and relative standard deviations RSDs (<20%) were achieved for the majority of the pesticides, except for chlorothalonil, which showed chaotic recoveries, and a few other pesticides with recoveries slightly outside of the range 70-120% at the low spiking levels (1-5 ng/g). The method was further validated by analysis of NIST Standard Reference Materials 1974B and 1947 for selected pesticides with certified concentrations. The measured values for both SRMs agreed with certified values (72-116% accuracy) for all pesticides, except for p,p'-DDD in SRM 1974B (45%) and for p,p'-DDT in SRM 1947 (52%). The developed method is fast, simple and inexpensive with the calculated sample preparation cost of ≈$2.5/sample using bulk materials. The developed method was successfully applied for analysis of fish samples from the market.

P15 Evaluation of Pesticides Contamination in Guarani Aquifer Recharge Area: GIS as Tool for Area Selection and Validation of Chromatographic Method

Paulo Alexandre Toledo Alves[1], Franz Zirena Vilca[1], Robson Rólland Monticelli Barizon[2] and Valdemar Luis Tornisielo[1]
[1]**Center of Nuclear Energy in Agriculture CENA**. University of São Paulo. Av. Centenário 303 CEP 13400-970, PO Box 96. Piracicaba São Paulo, Brazil; ptoedo@cena.usp.br;
[2]Embrapa Environment, Rd. SP 340 - Km 127,5 PO Box 69, Jaguariúna São Paulo, Brazil

The Guarani Aquifer (GA) is the largest underground water reserve in the world, but is under constant threat of contamination due to the intense use of recharge areas for agriculture. The intensive use of pesticides can lead to contamination of surface water, subsurface and groundwater, possible reaching aquatic organisms and humans. Other studies have been showing groundwater contamination by pesticides causing contamination and bioaccumulation in higher trophic levels. Given the current lack of coordination of the countries that have areas of recharge of the GA, it becomes very important to study the occurrence and assessment of contamination by pesticides, supporting statement regulatory agencies and the population for a healthy environment. The area for this study was chosen by GIS using as parameters the recharge area on the map, the land slope, soil type (sandy soil selected), soil depth, soil usage and hydrographic maps. The study will monitor pesticides contamination in the area during one year, analyzing subsurface water above soil, lake surface water and sediment and fish in a recharge area used for intensive agriculture in Brazil, Municipality of São Pedro, state of São Paulo. For validation the extraction technique SPE was used to extract the analytes from water samples, dispersive solid phase extraction (dSPE) for the sediments and QuEChERS (Quick, Easy, Cheap, Effective, Rugged) for the fishes samples. Fifteen compounds were used in the multiresidue methods, quantifications by LC-MS/MS. The recoveries were between 70-120% (RSD<20%) at 3 spiking levels. All calibration curves showed correlation coefficients greater than 0.99.

P16 Brazilian strategies for monitoring of POPs of Stockholm Convention

Paulo Alexandre Toledo Alves, Alberto da Rocha Neto, Camila Boechat, Cayssa Peres Marcondes, Lisandro Cogo Beck, Otávio Luiz Gusso Maioli, Álvaro Roberto Tavares, Marília Passos Torres de Almeida and Letícia Reis de Carvalho
Ministry of the Environment, Brazilian Federal Government, SEPN 505 Block B, Ed. Marie Prendi Cruz, T14, Brasília, Distrito Federal, Brazil; paulo.toledo@mma.gov.br

The Stockholm Convention was set in 2004 as one global agreement focused on control and elimination of use of Persistent Organic Pollutants (POPs). Since entered in force, all parties are pursuing its implementation applying the best efforts on chemicals management. Initially were settled 12 major substances (in great part organochlorine compounds as DDT and PCBs) and thereafter more 10 substances were included at COP (Convention of The Parties). Three main annexes to the text of the agreement (A, B and C) give the directions for treatments, elimination and control of emissions under the terms of the Convention. One of the principal subjects of these obligations is the monitoring of POPs in the environment as a tool for the evaluation of effectiveness of the implementation in each party. The POPs monitoring program in Brazil is coordinated by the Ministry of the Environment in partnership with CETESB and FIOCRUZ, and supported by the GRULAC (America Latina and Caribbean Group). The first step of the monitoring program was capacity building on analysis of POPs and analysis of matrices as urban air and human milk searching for POPs of great concern. Brazilian government is setting several projects on monitoring programs approaching research centers to the Ministry of the Environment with a GEF *Quick Start Project* for Stockholm Convention and also with own resources as counterpart. The next step for the POPs monitoring program is the establishment of one national wide monitoring network, using as examples the Arctic, the Canadian and the European initiatives.

P17 Evaluation of the Presence of Polycyclic Aromatic Hydrocarbons (PAHs) in the Neritic Areas for Populations of *Chelonia mydas* of the Brazilian Coast and Study of Residues in Individuals Affected by Fibropapillamitosis

Franz Zirena Vilca[1], Luana Eloisa Cezarino[1], Paulo Alexandre Toledo Alves[1], Roseli Gioia[2], Fabíola Prioste[2], Angélica Maria Sánchez Sarmiento[2], Silmara Rossi[2], Eliana Reiko Matushima[2] and Valdemar Luis Tornisielo[1]
[1]Center of Nuclear Energy in Agriculture CENA. University of Sao Paulo. Av. Centenario 303 CEP 13400-970, PO Box 96. Piracicaba Sao Paulo, Brazil, franzvilca@usp.br;
[2]Department of Pathology of the Faculty of Veterinary Medicine and zootechny of University of Sao Paulo – Brazil. Av. Prof. Dr. Orlando Marques de Paiva 87 CEP 05508-270 Sao Paulo, Brazil

The human activities have a strong impact on the marine environment, to the point that the viability of many ecosystems is threatened. The relationship between the oceans' health, anthropogenic activities and public health is a consensus in the scientific community; however, the mechanisms are still under scientific investigations. The presence of organic contaminants in marine compartments, such as polycyclic aromatic hydrocarbons (PAHs), has been reported by several researches in the world but its impact on the populations in these ecosystems is not fully understood. There binding of these contaminants is suspected in relation to the development of neoplasm in some animal, for example the fibropapillomatosis in species of sea turtles, problem that is endangering both threatened and endangered marine species. This project aims to assess the presence of PAHs in sediments, algae samples and adipose tissues or liver of sea turtles *C. mydas* with or not fibropapillomatosis from the Braziliam coast in order to better understand the origin of contamination of the turtles by these compounds, levels of contamination in these matrices, their spatial distribution of the contaminated areas and the possible quantitative relationship between PAHs in the development of this disease in *C. mydas*. The chromatographic methods and extraction methodologies were used for each matrix. For the validation process, samples of sediment, algae and liver of *C. mydas* with a low probability of PAHs residues were collected from Fernando de Noronha, which is an island located in the Atlantic Ocean and an area protected by Brazilian government.

P18 New Approaches to Trace Analysis of Pesticides in Biological and Environmental Samples

Maria Elena Y. Cabusas
DuPont Crop Protection, Stine-Haskell Research Center, 1090 Elkton Road, Newark, DE, USA; maria.e.cabusas@dupont.com

Trace level quantitative analysis of pesticides and/or their degradation products always poses a challenge. Widely varying properties of analytes (e.g., polarity, solubility, and stability) and the complexity of sample matrices make residue analyses difficult. When detection is by mass spectrometry, matrix effect problems are often encountered that purification steps are necessary. Typical residue methods involve purification by solid-phase extraction and analysis by HPLC/MS/MS. This presentation describes systematic approaches for rapid determination of residues by HPLC/MS/MS, without and with (off-line and on-line) SPE purification.

P19 Elimination of Matrix Effects and Interferences when Performing High Sensitivity and High Selectivity LC-MS/MS Screening

André Schreiber[1], Yun Yun Zou[1], Julia Jasak[2], Paul Yang[3], and Jon Wong[4]
[1]**AB SCIEX**, 71 Four Valley Drive, Concord, ON, Canada; andre.schreiber@absciex.com
[2]AB SCIEX, Darmstadt, Germany; [3]Ministry of Environment, Etobicoke, ON, Canada; [4]FDA-CFSAN, College Park, MD, USA

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 are a continuous challenge for food 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 and tea samples 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 Shimadzu UFLC$_{XR}$ system with a Restek Ultra Aqueous C18 column and a gradient of water and methanol with ammonium formate buffer. Total run time was less than 20 min. Detection was performed on the AB SCIEX QTRAP® 6500 system using Electrospray Ionization. In addition, SelexION ™ technology was used differential mobility separation (DMS) to enhanced selectivity to remove matrix interferences. 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. In addition, DMS was used to remove matrix interferences when detecting tricky to analyze (small molecular weight and high polarity analytes).

P20 Routine Targeted Quantitation and Identification of Pesticide Residues using QTRAP LC-MS/MS – The Use of Micro Flow UHPLC to Reduce Solvent Consumption

Stephen Lock[1], André Schreiber[2], and Brad Barrett[3]
[1]**AB SCIEX**, Phoenix House, Lakeside Drive, Warrington, UK; stephen.lock@absciex.com
[2]AB SCIEX, Concord, ON, Canada; [3]AB SCIEX Framingham, MA, USA

Traditionally in pesticide screening of food, samples are prepared using generic extraction procedures, like QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) and then analyzed by LC-MS/MS or GC-MS(/MS). Usually in LC-MS/MS analysis, LC flow rates exceed 400 µL/min and are used in combination with small particle size HPLC columns with high pressures to maintain sharp peaks and fast chromatography. These flow rates produce excellent peak shapes and results, but have a drawback in that they require higher volumes of organic solvents. The consumption of HPLC organic solvents, such as acetonitrile and methanol, is a growing cost of analysis, and their disposal can have an adverse environmental impact. Therefore, new approaches to reduce solvent consumption in pesticide residue testing will be beneficial to the environment while also reducing the running costs of a testing laboratory.

Food samples were extracted and analyzed using micro flow LC-MS/MS. LC separation was performed using an Eksigent ekspert™ micro200 system and targeted pesticides were detected using an AB SCIEX QTRAP® 4500 system using Electrospray Ionization. An enhanced *Scheduled* MRM™ algorithm was used to allow multi-component detection with best quantitative data quality and confidence in identification. At the same time the method provided huge cost savings of up to 90% because of reduced LC flow rate (40 µL/min) while maintaining robustness and reproducibility.

P21 Automatic Identification of Unknown and Unexpected Chemical Residues and Contaminants in Food Samples using Accurate Mass LC-MS/MS Screening Techniques

Lauryn Bailey[1], André Schreiber[2], David Cox[2], and Jeff Rivera[2]
[1]**AB SCIEX**, 500 Old Connecticut Path, Framingham, MA, USA; lauryn.bailey@absciex.com
[2]AB SCIEX, Concord, ON, Canada

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 pesticides, veterinary drugs, mycotoxins and other food residues. 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 of food contaminants. However, the use of triple quadrupole based mass analyzers is limited to targeted screening and quantitation. But there is an increasing demand for retrospective and non-targeted 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 QuEChERS procedure was used to extract residues and contaminants from fruit and vegetable samples. Extracts were subsequently analyzed by LC-MS/MS using an AB SCIEX TripleTOF® system operated in high resolution accurate mass MS and MS/MS mode. Full scan MS and MS/MS data was explored to identify known-unknowns using non-targeted data processing tools. Sample-control-comparison was successfully used to find unexpected contaminants. Identification was based on MS and MS/MS information, including 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 TripleTOF® software.

P22 Automatic Identification of Known Chemical Residues and Contaminants in Food Samples using Accurate Mass LC-MS/MS Screening Techniques

Farzad Pakdel[1], André Schreiber[1], David Cox[1], and Paul Yang[2]
[1]**AB SCIEX**, 71 Four Valley Drive, Concord, ON, Canada; farzad.pakdel@absciex.com
[2]Ministry of Environment, Etobicoke, ON, Canada

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 pesticides, veterinary drugs, mycotoxins and other food residues. 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 of food contaminants. However, the use of triple quadrupole based mass analyzers is limited to targeted screening and quantitation. But there is an increasing demand for retrospective and non-targeted 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 QuEChERS procedure was used to extract residues and contaminants from fruit and vegetable samples. Extracts were subsequently analyzed by LC-MS/MS using an AB SCIEX TripleTOF® system operated in high resolution accurate mass MS and MS/MS mode.

Full scan MS and MS/MS data was explored to identify knowns using extensive XIC lists of target compounds. Analytes were identified with high confidence based on retention time matching, mass accuracy, isotopic pattern, MS/MS library searching and elemental formula calculation based on MS and MS/MS ions. It was found that the use of MS/MS information is crucial to minimize false positive results. The latest revision of TripleTOF® data processing software makes this procedure intuitive and fast.

P23 Improved Sample Preparation Methods for Biophysical & Biochemical Characterization of Contaminants in Food by HPLC or LC-MS

Sam Ellis
Thomson Instrument Company, 1121 South Cleveland Street, Oceanside, CA 92054, USA; folks@htslabs.com

Modern HPLC, UPLC, GC/MS, LC/MS and other systems have revolutionized analytical chemistry by allowing biologists and chemists to do fast, cheap, and easy separations of complex mixtures. The real problem comes into play with the mixtures containing biological matrices and other debris blocking the equipment. The implementation of UPLC with the column particle sizes of less than 2 um (0.2 um inlet size frit) led to the increased system pressures by up to 300% and reduced tubing size to 1/32. Many analysts have seen the particulates clog up their system because of the changes. The Filter Vial is a simple, low-cost, upfront tool to saving time, expensive equipment, and maintenance headaches. It has allowed for the filtration process to become a SINGLE StEP® in the autosampler vial itself, eliminating the need for a syringe, syringe filter, HPLC vial and cap.

Which membranes are best for different applications? We have selected four membranes: Nylon, PVDF, PTFE, and PES. Nylon is a traditional filter technology but does not provide as good solvent resistance as PVDF. We found PVDF to have the lowest binding for pesticides and food contaminants in extracts with high water content (above 50% aqueous solutions). PTFE is a fully fluorinated or inert material that can be used for up to 100% organic, but due its hydrophobic properties should not be used for more than 50% aqueous solutions. PES is mainly employed for mammalian cell culture purification.

Patented Thomson eXtreme Filter Vials were made for viscous samples containing up to 30% solids. They were originally designed for the bioethanol analysis to eliminate multiple syringe filters and an expensive SPE step. We then worked with food labs analyzing juices, wine, coffee, and other matrices that had high pulp or other material content. This allowed them to press simply without hand stress that had occurred previously with syringe filters. Another important application area is protein analysis in meat, fish, and other high-protein matrices. This poster will demonstrate the use of eXtreme filter vials in the analysis of chemical contaminants in fish and other food matrices with a minimum sample preparation and without using any SPE clean-up step.

P24 Degradation of Aflatoxin B_1 by Fungal Isolates from *Meju*, a Fermented Starter for Soybean Paste and Soybean Sauce in Korea

Sun Min Yang[1], Sung Min Cho[1], Dong-Ho Kim[2], and Soo Hyun Chung[1]
[1]**Department of Food and Nutrition, Korea University**, Seoul 136-703, Korea; chungs59@korea.ac.kr;
[2]National Agricultural Products Quality Management Service, Seoul 150-804, Korea

Biological degradation of aflatoxin B_1 (AFB_1) by fungal isolates was screened and examined in liquid culture and in cell free culture liquid (CL). Rapid reduction of AFB_1 was observed by TLC and HPLC detection during cultivation of three fungal isolates. AFB1-biodegrading fungi were identified to *Apergillus oryzae, Eurotium chevalieri*, and *Rhizopus oryzae*, respectively, by sequencing the internal transcribed spacer region of fungal rDNA. The isolates of *A. oryzae* and *R. oryzae* degraded more than 90% of AFB_1 (initial concentration: 40μg/ℓ) within 9 days, *E. chevalieri* reduced ca. 55% of AFB_1. CLs of the three fungal strains were prepared by filtration (0.45 um) and used to investigate AFB_1-biodegrading ability under different pH conditions. With CL from *A. oryzae* and *E. chevalieri* AFB_1 was effectively degraded at pH 3.5 compared to pH 5.5 and 7.5, while CL from *R. oryzae* showed higher AFB1-biodegrading activity at pH 7.5. A possible degradation product of AFB_1 was detected by liquid chromatography–mass spectrometry (LC-MS/MS), whereas the degradation product could not be determined using HPLC-fluorescence detection.

P25 Use of a New Dual-Layer SPE Cartridge for Extraction of Polynuclear Aromatic Hydrocarbons from Olive Oil

Katherine K. Stenerson, Olga Shimelis, Ken Espenschied, and Jim Brown
Sigma-Aldrich/Supelco, 595 Harrison Road, Bellefonte, PA 16823, USA; katherine.stenerson@sial.com

Olive oil can become contaminated with polynuclear aromatic hydrocarbons (PAHs) through exposure of the olives to pollution in the

environment. Concern over exposure to these compounds has caused some countries within the European Union to set limits on PAH content in olive oil. In 2005, European Union Commission Regulation No 2008/2005 set a maximum limit of 2 ng/g PAH measured as benzo[a]pyrene in edible oil. Since that time, there has been discussion of monitoring additional PAHs.

Analysis of oily/fatty samples presents an analytical challenge due to the heavy matrix effects often encountered. In the case of GC-MS, fatty matrix can cause contamination of the GC inlet, column and detector. Various cleanup techniques exist for fatty samples, including gel permeation chromatography, liquid/liquid extraction, and solid phase extraction (SPE). The technique chosen for cleanup often depends on the analytical technique, specific target analytes, and required detection levels.

An SPE cartridge containing two different sorbent layers was evaluated in the simultaneous extraction and cleanup of PAHs from olive oil. The layers consist of Florisil and a mix of Z-Sep/C18. Olive oil sample was loaded directly onto the SPE cartridge, followed by elution of the PAHs with acetonitrile while fatty matrix remained bound to the sorbents. The resulting extract was concentrated, and analyzed by GC-MS. The dual-layer SPE cartridge was evaluated with olive oil samples spiked with 28 different PAHs, and found to yield recoveries of >70% for most compounds. In addition to being fast and convenient, the SPE method was found rugged and applicable to the GC-MS analysis of PAHs.

P26 Fast Extraction and Sensitive Detection of Low Levels of Chloramphenicol in Shrimp using Z-Sep+ Sorbent in QuEChERS Sample Preparation Approach and Analysis by LC-MS/MS.

Emily R. Barrey, Olga I. Shimelis, and Jim Brown
Supelco/Sigma-Aldrich, 595 N. Harrison Rd., Bellefonte, PA 16823, USA; olga.shimelis@sial.com

Chloramphenicol is a broad-spectrum antibiotic that has been used to treat several disease conditions in domestic animals and farmed fish. However, due to its potentially harmful side effects in humans, such as aplastic anemia and hypersensitivity reactions, it has been banned from use in animals for food production. In many countries, the tolerance for chloramphenicol in food products is zero. Fishery and aquaculture products are usually subject to tests to ensure the absence of chloramphenicol residues. The low detection limit of 0.1-0.3 ug/kg is set for chloramphenicol in shrimp.

Analytical confirmatory methods for chloramphenicol usually include time-consuming cleanup steps prior to LC-MS/MS analysis. Recently, new SPE sorbents, Z-Sep and Z-Sep+ became available. These were applied previously to preparation of fish tissue samples for analysis of pesticides, flame retardants and PAHs in a fast sample extraction and cleanup approach called QuEChERS. Using a QuEChERS like approach, both Z-Sep and Z-Sep+ sorbents were tested for the cleanup of extracted shrimp samples prior to analysis of chloramphenicol by LC-MS/MS.

The presented work will detail the fast sample preparation method using Z-Sep+ sorbent that resulted in the best overall recoveries and method performance. The sample cleanliness was compared to that from the “dilute-and-shoot” approach and found to be superior when using Z-Sep+ cleanup. The cleaner samples resulted in better method ruggedness, less instrument maintenance and better analytical performance. In addition, low quantitation limit of 0.1 ng/kg was easily reached by the proposed method.

P27 Emerging Contaminants in the Environment and in Drinking Water

Mark J. Benotti
Battelle Memorial Institute, 397 Washington Street, Duxbury, MA 02332, USA; benottim@battelle.org

The presence of emerging contaminants in the environment and in drinking waters has generated much of discussion about what, if any, toxicological significance they pose. To compliment this ongoing discussion, this poster will present results from several studies to highlight 1) emerging contaminant occurrence in the environment and drinking water, 2) the efficacy of conventional and advanced forms of water treatment and 3) novel approaches for elucidating the transformation products that are formed from some of these treatment technologies.

Nanogram-per liter (part-per trillion) concentrations of emerging contaminants, including some current-use pesticides, are found in aquatic systems and downstream drinking waters throughout the United States. The individual chemicals that persist in the environment are resistant to environmental processes like microbial degradation or photo-oxidation. The individual chemicals that persist in drinking waters are a function of the type of chemical oxidation used during the treatment process: ozone is a more effective oxidant than chlorine.

Identifying transformation products produced following water treatment is important given that they may bear some toxicological significance, and scientists have begun to investigate their occurrence. To illustrate this approach, the transformation of four compounds commonly detected in drinking water (atrazine, carbamazepine, diclofenac, and sulfamethoxazole) by low- and medium-pressure UV and UV-H_2O_2 will be discussed. Experiments were conducted with a collimated beam apparatus and samples were analyzed by both LC-MS/MS and QToF-MS approaches. In each case, disappearance of the target analyte occurred concomitantly with the appearance of transformation products.

P28 Sample Custody and Sample Identification Best Practices

Mark J. Benotti and Rosanna L. Buhl
Battelle Memorial Institute, 397 Washington Street, Duxbury, MA 02332, USA; benottim@battelle.org

Environmental field sampling is one step in an extensive process to collect new data for environmental assessments or compliance monitoring. In order to assure quality of data, there are a number of important activities that must be carefully executed. First, samples must be handled properly. This includes proper sample, storage, labeling, preservation, processing, and shipping. Next, sample handling and documentation must meet laboratory needs for acceptability. Finally, documentation must be sufficient to ensure that samples are traceable from field records to final reported data. It is also important to keep the end use in mind: how will data be managed, analyzed and reported? This poster will illustrate best practices for each of these elements. It will explain the reasons each element is important and identify problems that may result when errors occur. The requirements of the 2013 Department of Defense draft final Quality Systems Manual are specifically incorporated but the presentation also draws on Battelle's considerable field experience with the requirements of other organizations.

P29 Validation of a multi-residue method for determination of pesticides in lettuce using QuEChERS and LC-MS/MS.

Fernanda R. Begnini and Isabel C. S. F. Jardim
Institute of Chemistry, State University of Campinas, PO Box 6154, 13084-971, Campinas - SP, Brazil; ferbenini25@hotmail.com

According to the Brazilian Agency ANVISA, in 2010 lettuce showed irregularities in 54.2% of the samples analyzed, due to the presence of pesticides at levels above the maximum residue level (MRL). Therefore, the development of a multi-residue method for determination of pesticides in lettuce becomes necessary. For this, the quantification of pesticides was realized using QuEChERS for extraction and liquid chromatography-tandem mass spectrometry with a triple quadrupole mass analyzer and electrospray ionization (ESI) in the positive ion mode, previously optimized for the chosen pesticides. The pesticides extraction was performed using an optimized QuEChERS method, employing acetonitrile as extraction solvent, citrate buffer as partitioning salts, centrifugation at 5000 rpm for 5 minutes and clean-up with PSA, $MgSO_4$ and graphitized carbon black. The developed method was validated in terms of selectivity, detection and quantification limits, linearity, accuracy (recovery) and precision. The limits of detection of the method were in the range between 1.1 and 15.2 $\mu g\ kg^{-1}$ and the limits of quantification (LOQ) were from 5 $\mu g\ kg^{-1}$, established as a fraction of the MRL value, according to the *Codex Alimentarius* guidelines. The LOQ were lower than MRL for all pesticides. Linearity was evaluated in 5 levels of fortification and the concentration range and LOQ were different for each pesticide. The coefficients of determination were higher than 0.994, with residuals ≤ 10%. The method gave recoveries from 70 to 120%, in two levels of fortification, and a relative standard deviation ≤ 20% for all pesticides, in relation to repeatability and intermediate precision.

P30 Application of a Validated Multi-Residue Method for Quantification of Pesticides in Lettuce Samples from Brazil using QuEChERS and LC-MS/MS.

Fernanda R. Begnini and Isabel C. S. F. Jardim
Institute of Chemistry, State University of Campinas, PO Box 6154, 13084-971, Campinas - SP, Brazil; ferbenini25@hotmail.com

A multi-residue method for quantification of 16 pesticides in lettuce was developed and validated, since the presence of pesticides in levels above the maximum residue level (MRL) has been detected in this vegetable, according to the Brazilian Health Surveillance Agency (ANVISA). This method employed QuEChERS for extraction and liquid chromatography-tandem mass spectrometry with a triple quadrupole mass analyzer and electrospray ionization (ESI) in the positive ion mode, previously optimized for the chosen pesticides for identification and quantification. Samples of green leaf lettuce were purchased from different commercial market in Campinas, SP, Brazil, having been grown using two different types of management: soil and hydroponic. Each sample was separated into the leaves (with or without water washing) and the stalks, which were analyzed separately. The samples were submitted to extraction through the QuEChERS method, employing acetonitrile, citrate buffer and clean-up with PSA, $MgSO_4$ and graphitized carbon black. The quantification of pesticides was realized employing the matrix-matched standard solutions, in six levels of fortification, chosen as a function of the limits of quantification previously determined for each pesticide. In one sample analyzed, cultured in soil, methamidophos, acephate and carbendazim were detected in higher concentration in leaves than in the stalk. In relation to the samples cultured in hydroponic system, two of the three samples analyzed presented imidacloprid in levels higher than the MRL. Besides this, methamidophos, acephate and tebuconazole were also detected and quantified in these samples. These results indicate the need to continue to monitor lettuce.

P31 Risk Assessment of the Herbicide Glyphosate Use in Rice Crop in Saldaña, Colombia

Martha Cristina Bustos-López[1], Cilia Fuentes[2], and Gustavo Peñuela[3]
[1]**Civil and Agriculture Department, Universidad Nacional de Colombia**, Cra 30 No. 45-03, Bogotá, Colombia; mcbustosl@unal.edu.co;
[2]Agronomy Faculty, Universidad Nacional de Colombia, Bogotá, Colombia;
[3]Engineering Faculty , GDCON, Universidad de Antioquia, Medellin, Colombia

In rice crops in Saldaña (Tolima, Colombia), the glyphosate is used as weed control more than 30 years ago and in the last years, rice crop become a monoculture. A methodological proposal to evaluate the pesticides risk assessment in agriculture systems, where, integrating the primary and secondary information to estimate in different environmental compartments, estimate the risk levels which can be exposed the indicators organisms (earthworm, rats, bees, poultry, algae, fish and Daphnia), sensible to the pesticides. The first indicator is the hazard quotient (HQ) which considers the worst scenario. The second indicator is the ecological risk for pesticide application, RECAP which integrates the hazard in water, soil and air. The third indicator is the Risk Residues Index that estimates the risk in the food.

In this study, the DT50 of glyphosate was 360 days, then it is consider persistent, and the risk assessment give a chronic risk in epigeous and hypogeous terrestrial ecosystems, and an acute risk of toxicity for aquatic ecosystems. These results are contrasting with other recognized results stating that the risk for aquatic organism is insignificant at the same doses. In this risk assessment only is considered the glyphosate, but the transformation product AMPA (Amino methyl phosphonic acid) is present too in the environment, and other pesticides. With the actual information, is known that the glyphosate with other pesticides can provide a synergic effect, and increase the toxic effects over the biological activity in the soils, and the persistence of each individual compounds.

P32 Screening of pesticides and emerging contaminants in an agricultural area in Colombia by TOF MS coupled to gas and liquid chromatography

Martha Cristina Bustos-López[1], Tania Portolés[2], María Ibáñez[2], Felix Hernández[2], Cilia Fuentes[3], and Gustavo Peñuela[4]
[1]**Civil and Agriculture Department, Universidad Nacional de Colombia**, Cra 30 No. 45-03, Edif. 406, Of.325, Bogotá, Colombia, mcbustosl@unal.edu.co;
[2]Research Institute for Pesticides and Water (IUPA), Universidad Jaume I, Castellon, Spain ;
[3]Agronomy Faculty, Universidad Nacional de Colombia, Bogotá, Colombia; [4] Engineering Faculty , GDCON, Universidad de Antioquia, Medellin, Colombia

In a regional survey, water and soil samples from the irrigate district of Ususaldaña, in Tolima, Colombia were collected. GC-TOF MS was used for target screening of around 150 compounds (PAHs, PBDEs, alkylphenols, organochlorines, organophosphorous pesticides, triazines, pyretroids, etc). The screening was performed by automatically obtaining, at expected retention time, microwindow extracted Ion Chromatograms (mw-XIC), with a mass window of 0.02 Da, at selected m/z ions for every compound. Positive findings of atrazine, desethyl-atrazine and naphthalene were detected in water samples and p,p ’-DDD and p,p ’-DDE were found in soils. Additionally, a non-target analysis was performed by using Chromalynx Application Manager. Formulae from the library hit were submitted to elemental composition calculation and up to five ions were scored by exact mass measurement for confirmation/rejection of the finding. Several compounds, like clomazone, oxadiazon, butachlor, benzophenone, cadalenem calacorene, epizonarene, 1,6-dimethylnaphtalene, methylnapthalene, BHT, BHT-CHO, and N-BBSA were found by using the non-target approach.

UPLC-Q TOF MS data were processed using a compound database of over 1000 organic contaminants (pesticides, antibiotics, pharmaceuticals, illicit drugs and some of their metabolites, among others). Confirmation of the identity was performed using accurate masses, isotopic Fit (i-Fit) and retention time, as well as fragmentation information when available. Positive findings of atrazine, desisopropylatrazine (DIA), carbendazim, diuron, azoxystrobin, dimethazon, epoxiconazole, terbutylazine, thiacloprid, diniconazol, merphos, cyanazine, linuron and propiconazole were found in the different environmental matrices analyzed. The combination of GC and LC coupled to TOF MF allowed the investigation of a large number of contaminants with a wide range of polarities. Of the compounds detected, the majority are used in agricultural activities associated to rice crops.

P33 Automated GPC with Inline SPE to Improve Sample Cleanup for PAH Analysis

Jeffrey Wiseman, Leticia Brown, and Jennifer Salmons
J2 Scientific, LLC, 1901 Pennsylvania Drive, Suite C; Columbia, MO 65202, USA; jwiseman@j2scientific.com

Gel Permeation Chromatography (GPC) is a universal cleanup technique for environmental and food extracts that effectively separates high molecular weight compounds from analytes of interest including semivolatiles and pesticides. GPC alone is not always adequate to remove all of the high molecular weight compounds from particularly oily samples, and cannot always remove all compounds that interfere with the analytes in the determinative method. Solid Phase Extraction (SPE) can be used to remove these additional interferences, but additional cleanup can be labor-intensive and increases the risk of analyte loss with each step.

The automated system used for this study features SPE inline with GPC cleanup, allowing some or all of the GPC collect fraction to pass through one or more SPE cartridges or disks prior to concentration of the final sample. Performing SPE inline with GPC can provide additional cleanup without significantly increasing the processing time for each individual sample as the solid phase extraction is performed concurrently with the GPC collect fraction. The direct elution from the GPC column to the SPE cartridge also minimizes sample loss during the transfer. This presentation demonstrates the flexibility of the PrepLinc™ inline SPE option by showing recovery results for multiple classes of analytes, including PAHs and organochlorine pesticides, in a variety of matrices.

P34 Maximizing Lipid Load Without Chlorinated Solvents with 2-Dimensional GPC Cleanup

Jeffrey Wiseman, Leticia Brown, and Jennifer Salmons
J2 Scientific, LLC, 1901 Pennsylvania Drive, Suite C; Columbia, MO 65202, USA; jwiseman@j2scientific.com

The current EPA Gel Permeation Chromatography Cleanup procedure (Method 3640A) requires 300mL of methylene chloride for each sample processed, and the maximum recommended lipid load for a 5mL injection onto the traditional GPC cleanup column is 1 gram. Many laboratories are under pressure to replace their use of chlorinated solvents, and GPC columns with alternate solvents are available, but typically have a reduced lipid capacity.

The PrepLinc GPC-MAXX feature allows the user to control the GPC Dump, Collect and Wash steps through two columns in series, increasing the lipid capacity. A high-lipid sample can be injected onto the GPC with the first Dump to waste passing only through the first column. When the majority of the lipid has been removed from the sample, the second GPC column is switched inline to complete the separation. The lipid loaded onto the second column is reduced by the first column, maximizing separation in a single sample injection. Traditional GPC cleanup methods require large volumes of chlorinated solvents to clean up each individual sample. This study demonstrates the effectiveness of 2D GPC combined with alternate solvent systems to maximize lipid load without using chlorinated solvents.

P35 Analysis of Phthalates as Environmental Contaminants in the Quinnipiac River in Wallingford, Hamden, and New Haven, Connecticut

Harry M. Pylypiw, Jr. and Dipesh Khanal
Quinnipiac University, Department of Chemistry and Physical Sciences, 275 Mt Carmel Ave, Mail Stop BC-SCI, Hamden, CT 06518, USA; harry.pylypiw@quinnipiac.edu

The Quinnipiac River arises in central Connecticut and flows directly into New Haven Harbor, an inlet of Long Island Sound. In the 19th and 20th centuries, the river was severely polluted, and by 1914 was largely devoid of fish. Today, fishing is back, and the river is used widely by the public, but pollutants still exist in the form of phthalate-based plasticizers, which are used in the manufacturing of plastics. Characterized for their carcinogenic and teratogenic properties, studies have shown that these compounds are associated with reproductive effects in rats and fish even at low exposure. Research at Quinnipiac University has confirmed the presence of phthalate compounds at multiple sites along the Quinnipiac River near manufacturing companies which not only produce these compounds but which also have permits from the Connecticut Department of Energy and Environmental protection to discharge waste into the river. Analyses of water samples were performed via extraction with solid phase micro-extraction cartridges followed by detection with gas chromatography with a mass selective detector for compound identification. Climatic parameters (depth, rate of flow of water, ambient and water temperature) were also measured at the sampling sites. The phthalates, diethylhexyl phthatate, benzyl butyl phthalate and dibutyl phthalate were found to be present in the samples tested at levels exceeding EPA limits for drinking water, thus indicating a possible environmental risk to the surrounding ecosystem. Further research investigating the effect of phthalates in aquatic animals such as fish and crayfish are currently being conducted.

P36 Analysis of Systemic Pesticide Imidacloprid, and its Metabolites in Pepper using QuEChERS and LC-MS

Taek Kyum Kim, Hye Young Kwon, Su Myung Hong, Ji Hyeong Kwon, Eun Kyung Seo, and Doo Ho Kim
RDANational Academy of Agricultrual Science, 150 Suin-ro, Gwonseon-gu, Swon-si, Gyeonggi-do,441-707, Korea; ktkiii@korea.kr

Imidacloprid is systemic insecticides which acts as an insect neurotoxin. It is used for control of pests such as aphids and other sucking insects in fruits and vegetables. Systemic pesticides move inside a crop following absorption by the plant, and these are converted into a variety of metabolites. Sometimes these metabolites are problematic in terms of safety of agricultural products. Therefore, a simultaneous determination method for imidacloprid and its metabolites is needed, to monitor their presence in agricultural product and study on the fate of pesticide in a plant. The aim of this study was to investigate simultaneous analysis methodof imidacloprid and its metabolites imidacloprid urea, imidacloprid olefin, imidacloprid guadine, and 6-chloronicotinic acid in pepper using QuEChERS method and LC-MS systems. They were extracted by acetonitrile with 0.1% formic acid, and the extracts were purified through QuEChERS with primary secondary amine (PSA), and C_{18}, and analyzed with LC-MS in ESI positive mode. Standard calibration curves were made by matrix matched standards, and their correlation coefficients were higher than 0.998. Recovery studies were carried out on spiked pepper blank sample at three concentration levels (0.01 and 0.025, 0.1 mg/kg). The recoveries of imidacloprid and its metabolites were in the range of 70-120 % with <20% RSD. This result indicated that the method using QuEChERS and LC-MS was suitable for the simultaneous determination of imidacloprid and its important metabolites in pepper.

P37 Routine Trace Level Screening for a Broad Range of Organic Pollutants in Environmental Water Samples using Full Scan GC/MS with a >1000 Compound Target MS Database and Deconvolution Reporting Software

Wayne Civil[1], Chris Sandy[2], and Paul Stephens[2]
[1]**National Laboratory Service**, Environment Agency, Starcross, Exeter, Devon, EX6 8FD, UK
[2]Agilent Technologies UK Ltd., 610 Wharfedale Road, Winnersh, Berkshire, RG41 5TP, UK; chris_sandy@agilent.com

This poster details the use of a standard Agilent Technologies 7890/5975 GC/MSD system for the screening of GC-amenable organic contaminants in environmental water samples ranging from relatively 'clean' bore-hole waters and river waters to contaminated landfill leachates. The method employs a retention time locked method and a unique, application specific, EI MS database that includes the mass spectra of > 1000 organic pollutants including VOCs and SVOCs. Target components are identified by the ratio of a quantitation ion to three qualifying ions and also by library searching the full deconvoluted component mass spectra against the MS database, using retention time matching and library match quality as qualifiers in order reduce / remove false positives. Agilent's Deconvolution Reporting Software (DRS) processes the data for each data file in typically less than 2 minutes and combines the quantitative and qualitative results in to an easy-to-interpret report. This application was originally developed and designed for use with GC/MSD Chemstation software but can now also be run by employing the Target Deconvolution feature of the latest revision of Mass Hunter Quant software B.06.00 which was released in March 2013.

P38 New Levels of Mass Spectral Selectivity for Pesticide Residue Analysis: GC/Q-TOF in the MS/MS Mode with Chemical Ionization

Philip L. Wylie
Agilent Technologies, 2850 Centerville Rd., Wilmington, DE 19808-1610 USA; phil_wylie@agilent.com

Instrument selectivity has always been an important factor for the gas chromatographic analysis of pesticide residues. Even the most sensitive detector can give poor detection limits when chemical noise interferes with the analysis. In the past element selective detectors such as the electron capture detector (ECD), flame photometric detector (FPC), and nitrogen phosphorus detector (NPD) were used to "see" various classes of pesticides in food extracts. These detectors were supplemented and then largely replaced by GC/MS in the scan or SIM modes. More recently, labs have converted to unit mass GC/MS/MS because of its very high selectivity and sensitivity, even for dirty QuEChERS extracts. A new instrument, the GC/Q-TOF potentially offers even greater selectivity with its accurate mass capability together with MS/MS. Even more selectivity should come from the use of chemical ionization techniques. This poster discusses the use of a GC/Q-TOF for the analysis of pesticide residues in food extracts with a focus on selectivity for pesticides over matrix components. Comparisons are made between single stage accurate mass TOF analysis and MS/MS experiments in the Q-TOF mode. Chemical ionization is considered for its ability to give higher abundances of high mass ions as precursors for MS/MS experiments.

P39 Determination of Pesticides Transfer Rates (%) from Dried Tea Leaves to Brewed Tea

Wendy Cheung and Jian Wang
Canadian Food Inspection Agency, 3650 36 Street NW; Calgary, AB T2L 1L2, Canada; wendy.cheung@inspection.gc.ca

This paper presents an UHPLC/ESI-MS/MS method to determine 172 pesticides in brewed tea and a study on pesticide transfer rates (%) from dried tea leaves to brewed tea. A brewing process was simulated to prepare the tea for the study. Pesticides were extracted from a brewed tea sample using a procedure known as QuEChERS (quick, easy, cheap, effective, rugged and safe). UHPLC/ESI-MS/MS quantification was achieved using matrix-matched standard calibration curves with isotopically labelled standards or a chemical analogue as internal standards. The calibration curves consisted of six points (0.4, 2.0, 8.0, 16.0, 24.0 and 40.0 µg/L as in sample) and the method was validated at four concentration levels (4.0, 12, 20.0 and 32.0 µg/L) in triplicate with five different brewed tea matrices on two separate days per matrix.

The method performance parameters that included overall recovery, intermediate precision and measurement uncertainty were evaluated according to a nested experimental design. Approximately, 95% of the pesticides studied had recoveries between 81 and 110%; 95% of the pesticides had intermediate precision ≤ 20%; and 95% of the pesticides showed measurement uncertainty ≤ 40%. From a pilot study of 44 positive tea leaves samples, incurred pesticides were determined for their transfer rates (%) from dried tea leaves to brewed tea. Each sample was analyzed in duplicate for both tea leaves and brewed tea samples. Pesticides have different transfer rates (%). Three commonly found pesticides in tea leaves are carbendazim, methomyl and imidacloprid, which have average transfer rates of 83%, 92% and 85% respectively.

P40 Residual Characteristics of Cyhalothrin, Fenitrothion and Flufenoxuron in Green Tea

Jeong-Hwan Song[1], Soon-Kil Cho[1], Yang-Mo Jeong[1], Jong-Hyouk Park[2], and Jae-Han Shim[2]
[1]**National Agricultural Products Quality Management**, Gwangju, Republic of Korea; sjhjerg@korea.kr;
[2]Chonnam National University, Gwangju, Republic of Korea

The purpose of this research is to figure out characteristic of pesticide residues in each phase of tea processing by investigating residues at the fresh leaf state, changes during the roasting process, and the amount of pesticides extracted by brewing (at different temperatures) at the final consumption phase after cyhalothrin, fenitrothion and flufenoxuron pesticide application on tea tree (Camellia sinesis.L). This experiment is to spray it after diluting cyhalothrin WP(a·i 1 %), flufenoxuron DC(a·i 5%) and fenitrothion WP (a·i 40%) to tea tree as a standard amount. Temporal changes of pesticides is looked into by analyzing residue of pesticides from spraying date (2 hours after spraying) to 10th day. After adding acetonitrile to chopped sample and doing homogenization, n-hexane is done as fraction from 10% NaCl aqueous solution through vacuum filtration and after cleaning it up to Florisil cartridge column, it is analyzed to GC/ECD. After repeating 3 times of roasting and rubbing fresh leaves in cauldron at 320℃±50℃, roasting beverages

is prepared as roasting sample. 2 hours after adding water to roasting sample, it is analyzed as the same method as fresh leaves. As immersing roasting sample in water of 60℃, 80℃ and 100℃ respectively, it is analyzed as GC/ECD by adding n-hexane to brewed water for 3 minutes and shaking centrifugation without refining process.

Fortified level for recovery test at each phase were 0.1 mg/kg and 1.0m g/kg and recovery rate of cyhalothrin at fresh leaves is 76.4±1.3%~76.8±1.3% and recovery rate of fenitrothion is 101.5±0.3%~105.6±0.6% and recovery rate of flufenoxuron is 99.1±0.7%~96.6±3.2%. Detection limit by GC/ECD was 0.01 mg/kg. At that day of spraying, level of cyhalothrin had gradually decreased from 0.35 mg/kg and it decreased to 0.12 mg/kg on the 10the day and fenitrothion decreased from 35.0 mg/kg to 0.2 mg/kg on the 10the day and flufenoxuron gradually decreased from 2.93m g/kg to 0.73 mg/kg on the 10the day. As moisture decreased through roasting process, cyhalothrin increased 4.0 times and fenitrothion increased 2.8 times and flufenoxuron increased 2.9 times. As a result on brewing roasting tea at 60℃, 80℃ and 100℃ temperature, cyhalothrin and flufenoxuron are not extracted at all (due to their low solubility in water). Fenitrothion was extracted 10.7% at 60℃, 19.4% at 80℃ and 26.6% at 100℃, thus the extraction rate of fenitrothion was increasing with the increasing brewing temperature.

P41 Automated Solid Phase Extraction (SPE)-LC/MS/MS Method for the Determination of Acrylamide in Brewed Coffee Samples

Oscar G. Cabrices, Fred F. Foster, John R. Stuff, and Edward A. Pfannkoch
GERSTEL Inc., Linthicum, MD, USA; ogcabrices@gerstelus.com

Acrylamide is thought to be produced during the roasting process associated with coffee production. Acrylamide has been labeled as a probable human carcinogen. Due to the use of roasted coffee beans in making coffee and the high consumption of coffee by people living in the United States and in many other countries, brewed coffee could be a source of daily exposure to acrylamide. Acrylamide determination has been shown to be one of the most challenging methods due to co-extractives that can exist in the final extract. Manual solid phase extraction followed by LC/MS/MS analysis has been reported as a successful method for the determination of acrylamide from brewed coffee samples. However, these solid phase extraction methods can be tedious and time consuming when performed manually. There is therefore an increasing need for the automation of solid phase extraction methods.

In this study, we show that a manual SPE method used for the determination of acrylamide in brewed coffee samples can be converted to an autosampler compatible cartridge format and automated using a robotic autosampler controlled by user-friendly software. Calibration standards prepared in freshly brewed green coffee (unroasted) resulted in a linear calibration curve (R^2=0.99) from 1ng/mL to 500ng/mL. Precision of the automated SPE-LC/MS/MS method was calculated as CV= 2%.

P42 Automated sample preparation and analysis workflows for pesticide residue screenings in food samples using DPX-QuEChERS with LC/MS/MS

Oscar G. Cabrices[1], Edward A. Pfannkoch[1], Fred F. Foster[1], Andre Schreiber[2], and William E. Brewer[3]
[1]**GERSTEL Inc.,** Linthicum, MD, USA; ogcabrices@gerstelus.com;
[2]AB SCIEX, 71 Four Valley Drive, Ontario, Canada;
[3]Department of Chemistry and Biochemistry, University of South Carolina, Columbia, SC, USA

One of the most important aspects of reducing pesticide exposure is to monitor for pesticide residues in foods due to the health risks they pose to humans and livestock. A number of analytical methods have been published, many of them based on traditional liquid-liquid extraction in combination with GC-MS or LC-MS. The QuEChERS (quick, easy, cheap, effective, rugged, and safe) sample preparation methods have been developed to help monitor pesticides in a range of food samples. These methods, however, still require many manual steps, such as shaking, mixing, centrifugation, and dispersive SPE, making it a quite labor-intensive process. There is a need for automating the dispersive SPE technique to clean up QuEChERS type extracts in order to improve laboratory productivity for monitoring pesticide residue in complex food matrices.

In this report, we describe an automated sample preparation and analysis workflow for the screening of pesticides residues in different food matrix (fruits, vegetables and nuts) by LC/MS/MS. The automated cleanup of the QuEChERS extracts methodology was performed using disposable pipette extraction (DPX). DPX is a solid-phase extraction (SPE) technique that is based on loosely contained cleanup sorbent inside a pipette tip fitted with a screen. Analytical methodology for confirming the presence of a variety of pesticides in various food samples was developed using a GERSTEL MPS robotic autosampler interfaced to an AB SCIEX QTRAP® 4500 LC/MS/MS System. Two transitions per parent compound were monitored using a single a *Scheduled* MRM™ method. The automated DPX-QuEChERS cleanup procedure provided extraction efficiencies greater than 70% for all pesticides 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) allowing detection limits of the method to meet acceptance criteria for reporting maximum residue levels (MRLs) as established by regulatory agencies. The ability to automate dispersive SPE clean-up of QuEChERS extracts and to couple the extraction directly to LC/MS/MS analytical methods, results in improved laboratory productivity by streamlining the complete analytical process.

P43 A Modified QuEChERS Approach for the Analysis of Pesticides in Fruit Juice Concentrate

Derick Lucas
Agilent Technologies, 2850 Centerville Road, Wilmington, DE 19808, USA; derick_lucas@agilent.com

As analytical laboratories are challenged with the task of meeting lower detection limits for an increasing number of compounds in a variety of matrices, the burden relies not only on instrument performance but also on efficient sample preparation. A modified Quick, Easy, Cheap, Effective, and Safe (QuEChERS) method was created to extract up to 70 pesticides from very challenging samples in terms of extreme pH (lemon juice concentrate) and texture (orange juice concentrate). Juice concentrate samples were provided by a worldwide beverage company in collaboration for method development. With these complex samples, the QuEChERS method was modified to the account for sample pH, volume, and chemistries to ensure effective matrix removal and improved recovery. Tandem GC-MS/MS was used for pesticide detection and quantification, and results will be presented for both lemon juice and orange juice concentrates.

P44 Optimizing Sample Preparation for Herbal Green and Black Teas

Derick Lucas
Agilent Technologies, 2850 Centerville Road, Wilmington, DE 19808, USA; derick_lucas@agilent.com

The popularity of botanical supplements and herbal commodities requires regulatory agencies to enforce maximum residue levels (MRLs) and accurate labeling practices. For these reasons, it is necessary for laboratories to have the appropriate analytical protocols to test these commodities for contaminants, adulterants, and other compounds of interest. Sample preparation is an essential step in these protocols as botanical matrices are often complex and contain interfering compounds that can lead to ion suppression and co-elution in LC-MS/MS analysis. This study implements the Quick, Easy, Cheap, Effective, and Safe (QuEChERS) technique in the preparation of black and green tea samples for the analysis of pesticide residues. The sample cleanup utilized adsorbent materials such as C18, primary secondary amine (PSA), and graphitized carbon black (GCB). The combination of adsorbent materials effectively removes unwanted components, while providing higher recoveries, lower detection limits, and improved instrument cleanliness. LC-MS/MS analysis was used for analyte detection and quantification.

P45 Improved MRM Confirmation for Multi-residue Pesticide Screening and Quantitation

Jerry Zweigenbaum
Agilent Technologies, Inc., 2850 Centerville Road, Wilmington, DE, USA; j_zweigenbaum@agilent.com

Screening of 100s of pesticides using LC/QQQ MS/MS in MRM mode has become the gold standard of multi-residue analysis. However, it has been long recognized that screening this many compounds using retention time, a quantifier transition and one qualifier transition could lead to misidentification. For example, under typical chromatographic conditions fenpropathrin and chlorpyriphos can co-elute. Using their nominal mono-isotopic protonated m/z for the precursor, the two most abundant nominal mass transitions for qualifier and quantifier are all the same and their ratios are also similar. A technique to address this problem adds additional qualifier transitions only when a threshold value is obtained for a primary transition being monitored. The additional confirmation transitions are collected only for a specified number of cycles and intervals. In this way pairs like the above cited compounds can be discerned and confirmed by comparison of a “pseudo spectrum” comprised of the additional transition to a “standard” library spectrum. Examples of different types of food samples (e.g. spinach/orange juice) will be used to demonstrate how this methodology can obtain a much higher degree of confidence in confirmation without sacrificing quantitative determination. Proper selection of parameters to obtain both quantitative and highly qualitative results in one analysis even when there are multi-coeluting compounds will be described. In addition, data will be shown that delineate the boundaries of using this methodology such as effect on sensitivity and quantitative results when multiple compounds co-elute in the standard but only one compound is present in the sample.

P46 Determination of Pesticide Residues on Organic and Non-Organic Fruits and Vegetables

Frank L. Dorman[1], Ashley Gates[1]; Christopher Solloway[1]; Elizabeth Humston-Fulmer[2]; Joe Binkley[2] and Jessica Westland[1]
[1]**The Pennsylvania State University**, 107 Whitmore, State College, PA 16802, USA; fld3@psu.edu;
[2] Separation Sciences, Leco Corporation, MI, USA

Pesticides, chemicals used to control or eliminate pests, are typically used in the agricultural industry to prevent crop destruction and loss. However, the pesticides often find their way into food purchased and eaten by consumers. As a result, many people purchase organic commodities to reduce the amount of potentially ingested pesticides, but do organic foods still contain pesticides? And if so, how do the pesticides found in organic commodities compare to those found in non-organic commodities? By employing a sample preparation technique, “QuEChERS” (“catchers”), pesticides can be extracted from both organic and non-organic fruits and vegetables that are unwashed, or have been washed with either a commercially available fruit and vegetable wash, or tap water. The European EN 15662 QuEChERS method determines the identity of pesticide residues using GC-MS following acetonitrile extraction of pesticides and dispersive solid phase extraction (dSPE), a technique utilized to remove sugars, lipids, organic acids, sterols, proteins, pigments, and excess water.

This experiment will demonstrate the effectiveness, efficiency and ease of using the QuEChERS extraction method and also determine the identity and amount of any incurred pesticides found in the food commodities. Discussion will be given to the strengths of the technique as well as to the significance of any pesticides detected. Finally, the potential benefits of different washing preparations, and the decision to purchase organic versus non-organic commodities will be summarized.

P47 Discovery-Based Analyses of Wastewater Samples for Determination of Emerging Contaminants

Frank L. Dorman[1], Adrienne Brockman[1], Jack Cochran[1,2] and Michelle Misselwitz[2]
[1]Pennsylvania State University, 107 Whitmore, State College, PA 16802, USA; fld3@psu.edu
[2]Restek, 110 Benner Circle, Bellefonte, PA 16823

Monitoring of wastewater treatment facilities (WWTF) may be used for a variety of purposes. The objective of this research is to develop an analytical strategy to determine emerging compounds of concern, including toxic compounds, bio-terrorism events, illicit drugs and drug metabolites. Specifically, wastewater samples obtained from the Pennsylvania State University wastewater treatment plant will be used as a control facility to refine analytical methodology. Rather than beginning with a target compound approach, a discovery analysis approach was chosen to try and determine as many compounds as possible prior to any compound list restriction. The difficulty in this approach can be the resulting complexity of the analysis. For this reason both Comprehensive Gas Chromatography coupled with Time-Of-Flight Mass Spectrometry (GCxGC-TOFMS) analysis and High Performance Liquid Chromatography coupled with Time-Of-Flight Mass Spectrometry (HPLC-TOFMS) analysis were utilized for their inherent ability to characterize these potentially complex samples more successfully compared to other possible techniques. The ultimate goal is to determine emerging contaminants and define temporal and spatial characteristics of usage at the community level.

Multiple four-liter samples were gathered from four different stages throughout the Penn State WWTF. Following USEPA method 3510c, a liquid-liquid extraction process was performed and Kuderna-Danish technique was used to concentrate the samples to 1 mL. Once the samples had undergone the clean-up process they were introduced to the analytical systems, to identify and quantify the present compounds. Equally challenging is the need to develop and define what is to be considered “normal” so that this background can be subtracted from subsequent samples in order to develop an approach that is capable of determination when a new “outlier” is detected

P48 Introducing the Q&A Handbook for Pesticide Residue Analysis by the Pesticide Society of Japan

Kazuaki Iijima[1] and Yoko S. Johnson[2]
[1]Institute of Environmental Toxicology, 4321, Uchimoriya-machi, Joso-shi, Ibaraki 303-0043, Japan; iijima@iet.or.jp;
[2]Minnesota Department of Agriculture, 601 North Robert Street; St. Paul, MN 55155, USA; yoko.johnson@state.mn.us

When a residue chemist comes across work related questions, where does he/she look for answers? When an experienced chemist new to the pesticide residue work is looking for a quick overview of the area, where does he/she go? Talking to co-workers, attending workshops, reading books and websites are some of the many options available. You may have felt that there are not many references that answer your questions from the perspective of pesticide residue chemistry. We are introducing a convenient, simple, Q&A format handbook that is dedicated to the area of pesticide residue analysis published by the Pesticide Society of Japan. It consists of about 100 questions with each answer succinctly summarized in less than 2 pages. The Q&A format makes it easy for readers to find pages that are related to their questions. Yet, categories are organized in a way that the handbook can be read through from the beginning to the end. Topics covered include: regulatory standards, analytical apparatus, sampling, extraction and cleanups, instrumental analysis, quality control including ISO 17025. Residue analysis in environmental matrices is also covered. By sharing the experience of using the handbook, the authors hope to emphasize the importance of and need for easy to use, up to date, and practical references in the area of the pesticide residue analysis.

P49 Applying Micro Flow LC and High Speed Data Acquisition MS/MS to the Analysis of Pesticides Residues in Complex Spice Matrix

David R. Baker[1], Neil Loftus[1], and Simon Hird[2]
[1]**Shimadzu**, Wharfside, Trafford Wharf Road, Manchester, UK, M17 1GP; david.baker@shimadzu-mso.com
[2]Food and Environment Research Agency, York, UK

In the UK, the Committee on Pesticide Residues in Food (PRiF) monitors levels of residues in food. PRiF adopts a risk-based approach to monitoring the foodstuffs most likely to contain residues. The 2012 survey included the analysis of spices, which are recognised as a challenging matrix. Spices are known as “difficult” matrices, since they contain a bulk of extractable compounds (essential oils, pigments, etc.) causing significant problems during LC–MS/MS analysis including loss of chromatographic performance and system robustness, matrix effects and isobaric interference. Clean-up usually limits the scope of target analytes, thus, an extensive clean-up is not practical when faced with multi-residue analysis. In this presentation we will present the application of micro flow LC in comparison to conventional flow LC to minimise the challenges associated with the analysis of spice extracts. An established pesticide method using a 2.1 mm ID x 100 mm column and a 400 µL/min flow rate was compared to a micro flow method. Linear velocity was maintained during the micro flow analysis on a 1.0mm ID x 100mm column by using a 90µL/min flow rate. Both methods were utilised for the analysis of 210 pesticides in spice extracts. A comparison of the conventional flow method and the micro flow method resulted typically in an increase in peak height of 2 to 3 times. The increased sensitivity afforded by micro flow LC was utilised to reduce injection volumes, which aided the reduction of matrix effects.

P50 Development and Validation of a Standardized Method for the Determination of Morpholine Residues in Fruit Commodities in Response to Import Restrictions

Rick Jordan[1], Matt J Hengel[2], and Wesley Maguire[1]
[1]**Pacific Agricultural Laboratory**, 12505 NW Cornell Road, Portland, OR 97229, USA; rjordan@pacaglab.com [2]Department of Environmental Toxicology, University of California, Davis, CA, USA

Morpholine is a food additive used in the process of glazing fruit, acting as an emulsifier for the wax coating applied to the surface of the fruit. The use of morpholine is approved in many countries worldwide, including the US, Canada, Australia, and Chile. In 2010, the UK Food Standards Agency (FSA) reported morpholine residues in apples imported from Chile to the UK. The use of morpholine as a food additive is prohibited under EC Regulation 1333/2008, as it is considered to be a precursor of the carcinogen N-nitrosomorpholine. As a result of this ban, imported fruit is subject to testing for morpholine residues prior to entering the marketplace. Fruit that tests positive for morpholine residues is sent back to the country of export at the shipper's expense.

To date, laboratories have used proprietary methods, and a standard peer reviewed and published method has not been available. This denies exporting countries the ability to determine the presence of morpholine residues using the same techniques as the importer prior to shipment.

Due to its high polarity, morpholine is not amenable to a traditional QuEChERS technique. An extraction for highly polar pesticides (QuPPe) has been developed, and coupled with analysis by LC-MS/MS can be optimized for the determination of morpholine residues. Samples are extracted using 1% acetic acid in methanol and water. The extract is analyzed directly by LC-MS/MS using a mixed mode column (Sielc Primesep A). Method validation was performed at 10 ug/kg, 40 ug/kg, and 200 ug/kg on both apple and citrus matrices.

P51 Development of a Multiclass Cleanup Method Incorporating a Novel SPE Media for the Analysis of Mycotoxins in Grain Using LC-MS/MS

Geoff Davies[1], Claire Desbrow[1], Steve Jordan[1], Frank Kero[3], Mats Leeman[2], Helen Lodder[1], Adam Senior[1], Kerry Stephens[1], Victor Vandell[3], and Lee Williams[1]
[1]**Biotage GB Limited**, Distribution Way, Dyffryn Business Park, Hengoed, CF82 7TS, UK; adam.senior@biotage.com; [2]MIP Technologies AB (a subsidiary of Biotage AB), Box 737, SE-220 07, Lund, Sweden;
[3]Biotage LLC, 10430 Harris Oaks Blvd., Suite C, Charlotte, NC 28269, USA

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

P52 Development of a Cleanup Method Incorporating a Novel SPE Media for the Analysis of Patulin in Apple Juice Using LC-MS/MS

Geoff Davies[1], Claire Desbrow[1], Steve Jordan[1], Frank Kero[3], Mats Leeman[2], Helen Lodder[1], Adam Senior[1], Kerry Stephens[1], Victor Vandell[3], and Lee Williams[1]
[1]**Biotage GB Limited**, Distribution Way, Dyffryn Business Park, Hengoed, CF82 7TS, UK; adam.senior@biotage.com; [2]MIP Technologies AB (a subsidiary of Biotage AB), Box 737, SE-220 07, Lund, Sweden;
[3]Biotage LLC, 10430 Harris Oaks Blvd., Suite C, Charlotte, NC 28269, USA

Patulin is a mycotoxin produced by Aspergillium and Penicillium fungal species commonly found on rotting apples. Although not a particularly potent toxin, patulin has been shown to be genotoxic and potentially carcinogenic, requiring regulation and analysis in apple-based products. Recommended maximum limits for patulin are set globally at 10 $\mu g\ kg^{-1}$ in apple juice, 25 $\mu g\ kg^{-1}$ in solid apple foods, and 10 $\mu g\ kg^{-1}$ in apple-based infant food. Mycotoxins are a structurally diverse group of toxic metabolites produced by several strains of fungi found on food crops. Their potential to cause harm to humans, crops and farmed animals means that a wide range of food matrices need to be tested for mycotoxin contamination. The diversity of both analyte structure and food matrix produces a significant analytical challenge. Traditionally, mycotoxins have been analyzed using multiple methods, each one optimized for a single mycotoxin or group of very closely related toxins. The increasing adoption of liquid chromatography-tandem mass spectrometry (LC-MS/MS) based analysis facilitates a multi-analyte approach. Here we present work on the development of simple catch-and-

release sample preparation method using a novel solid phase extraction (SPE) column for the determination of patulin contamination of apple-based products. The extraction method demonstrates high recovery of patulin whilst the accompanying HPLC method effectively resolves hydroxymethylfurfural, a significant endogenous interference. We are able to demonstrate patulin recovery of 101% and an LOQ of 10 µg kg^{-1} with an analytical working range of 2 to 200 µg kg^{-1} meeting regulatory requirements for patulin maximum residue limits.

P53 Using the Direct Analysis in Real Time Green Screening Interface (DART GSX) System for Accurate Mass Analysis of Pesticides

Joseph LaPointe, Brian Musselman, and Robert Goguen
Ionsense, 999 Broadway; Saugus, MA 01906, USA; lapointe@ionsense.com

Ambient ionization sources such as Direct Analysis in Real Time (DART) and Desorption Electrospray Ionization (DESI) were facilitated by the availability of high performance liquid chromatography/ mass spectrometry systems (LC/MS). Compared to gas chromatography MS (GC/MS) the number of these LC/MS systems in operation is relatively small.

DART, an ambient pressure ionization source, generates intact $[M+H]^+$ molecules by introducing the sample to a gas stream of metastable nitrogen molecules which can be heated to permit thermal desorption. In order to provide a more widely available platform for ambient ionization we have enabled an atmospheric pressure inlet for use with the Agilent 5973 series GC/MS along with DART. The instrument design includes a three stage vacuum system with capillary inlet and ion guide for optimum transfer of ions into the mass selective detector (MSD). The interface enables both DART and micro-electrospray ionization sources. Sensitivity of the device was measured using typical standards employed for DART. The modified GC/MSD system was utilized for the analysis of pesticides in herbal supplements. Using Massworks™ software (Cerno Biosciences) the normally nominal mass data can be converted to accurate mass data. The DART-GSX and Massworks software allows for rapid screening of contaminating samples by using accurate mass and validation compounds.

P54 An Engineered Carbon Material for Efficient Cleanup of Highly Pigmented Extracts

Conor S. Smith,[1] Jonathan D. Thompson,[1] Douglas Fryer,[1] and Dwight Stoll[2]
[1]**United Science Corp**, 15911 Furuby Rd; Center City, MN 55012, USA; csmith@unitedsciencecorp.com;
[2]Gustavus Adolphus College; St. Peter, MN

Sample preparation is of expanding importance as cleanup of new and more difficult matrices and analytes becomes important. Generally carbon sorbents are sourced from natural materials such as coconut shells or other waster material. This can introduce issues with reproducibility and flexibility. Currently graphitic carbon black (GCB) is used for pigment cleanup in QuEChERS, but has issues with adsorbing planar pesticides such as chlorothalonil and coumaphos. We are introducing a carbon sorbent that is engineered atop metal oxide frameworks, allowing for tunable carbon loadings and surface chemistries with extremely high mechanical strength. Due to the well ordered core materials the surface areas, flow rates, and particle size distributions are much tighter than the currently used GCB. The sorbent CarbonX Plus has been optimized for QuEChERS cleanup of highly pigmented extracts including vegetables such as spinach and red pepper, as well as botanicals such as saw palmetto and ginkgo biloba. In addition, it has excellent recoveries of planar pesticides without the need for alterations to the standard EN and AOAC QuEChERS cleanup methods.

P55 Rapid Detection of 250 Pesticide Residues in Okra Using Ultra Performance Liquid Chromatography and Tandem Mass Spectrometry

Dimple Shah[1], Mark Benvenuti[1], P.M.N. Rajesh[2], Antonietta Gledhill[3], and Jennifer A. Burgess[1]
[1] **Waters Corporation**, 34 Maple St, Milford, MA 01757, USA; jennifer_burgess@waters.com
[2]Waters India, Peenya Industrial Area, Bangalore, 560 058, India;
[3] Waters Corporation, Atlas Park, Manchester, M22 5PP, UK

Okra is an important vegetable of the tropical countries and most popular diet component in different countries including India. According to the Food and Agriculture Organization of the United Nations (FAO), India is the second largest producer of this vegetable in the world and it produced approximately 5,800 tones of okra in 2010-11. Okra is susceptible to a variety of pests and diseases. A wide range of pesticides are used to treat okra plants in India. Legislative limits are in place for the presence of pesticides in domestically produced, imported, or exported okra. It is, therefore, very important to monitor commonly used pesticides at legislative limits.

A multiresidue analysis method for the detection of 250 pesticides in okra will be presented. Samples were extracted using either acetate or citrate buffered QuEChERS methods. The extracts were subjected to a dispersive solid-phase extraction using different combinations of MgSO4, PSA, GCB and C18 to determine the most appropriate method to further clean the okra extracts prior to analysis. Ultra performance liquid chromatography coupled with tandem quadrupole mass spectrometry with 2 MRM transitions per compound were collected for all pesticides in either ESI+ or ESI- mode using rapid polarity switching. Simultaneous full scan data was acquired in order to assess any matrix effects. Product ion confirmation scans were also acquired simultaneously to confirm pesticide identifications. For all pesticides, limit of detection (LOD), limit of quantification (LOQ) and recoveries at 10 µg/kg will be presented.

P56 Multiresidue Analysis of Pharmaceuticals and Personal Care Products in Water

Claude Mallet, Joseph P. Romano and Jennifer A. Burgess
Waters Corporation, 34 Maple St., Milford, MA, USA; joe_romano@waters.com

Concern regarding the presence of pharmaceuticals and personal care products (PPCPs) in water bodies throughout the world has increased in recent years. PPCPs refer to any chemicals used for human health, cosmetic and agribusiness (growth/health of livestock). The list is vast and includes prescription medication, over-the-counter drugs (OTCs) and veterinary drugs as well as compounds present in fragrances, lotions and cosmetics. The effect of these emerging contaminants on human health and their potential impact on the environment is not yet fully understood. As this concern continues to grow, many government agencies around the world are funding studies to assess if PPCPs can cause harmful ecological effects.

A major analytical challenge for the analysis of PPCPs is their wide chemical diversity, encompassing many compound classes and structures. In addition, the complexity of the water samples requiring analysis can be very diverse. It is therefore critical to ensure a robust, reproducible multi-residue LC-MS/MS protocol is employed. This work demonstrates the extraction, separation and detection of 83 PPCPs including acidic, basic and neutral compounds. Water samples from bottled, tap and surface water were extracted using mixed-mode SPE with two connected cartridges. Combined extracts were analyzed on a 2.1 x 100 mm HSS T3 analytical column (1.7um) using methanol/water with 10 mM ammonium formate (pH 3.2). Two MRM transitions (quantification and confirmation) for all PPCPs were monitored. Results show the capability of detecting ultra trace levels (parts per trillion) of these compounds in water samples using a small-footprint, routine, benchtop tandem quadrupole mass spectrometer.

P57 A Modified QuEChERS Extraction, Simple Silica Cartridge SPE Cleanup, and Hydrogen Carrier GC-TOFMS and GC-MS/MS with a Novel Selectivity GC Column to Determine the EFSA PAH4 in Mate and Other Teas

Julie Kowalski[1], Amanda Rigdon[1], and Jack Cochran[1,2]
[1]**Restek Corporation**, 110 Benner Circle, PA 16823, USA; julie.kowalski@restek.com
[2]The Pennsylvania State University, University Park, PA, USA

Polycyclic aromatic hydrocarbons (PAHs) are toxic compounds found in some foods, especially those that are smoked, roasted, grilled, or dried during preparation. Teas, including yerba mate, contain PAHs, sometimes at relatively high levels. While classic sample preparation methods such as Soxhlet and Pressurized Fluid Extraction (PFE) yield excellent quantitative results for PAHs in tea, Soxhlet has high solvent use and PFE requires expensive capital equipment. The more simple QuEChERS procedure requires little solvent and no expensive equipment, so it is a natural choice for consideration as a replacement extraction method for PAHs in tea.

We used a modified QuEChERS procedure for extraction of PAHs from teas. Acetonitrile, the classic QuEChERS solvent, was inefficient at extracting PAHs from the complex tea matrix, but hexane:acetone (50:50) gave quantitative recoveries. A simple silica cartridge cleanup with one elution solvent combination was used to clean extracts, including the removal of chlorophyll that can foul GC inlets and columns. A novel GC stationary phase with selectivity towards PAHs separated EFSA PAH4 benz[a]anthracene, chrysene (triphenylene was separated), benzo[b]fluoranthene (separated from other benzo fluoranthenes), and benzo[a]pyrene under hydrogen carrier GC-TOFMS and GC-MS/MS conditions. A candidate NIST SRM mate tea was analyzed for PAHs with our method in addition to characterizing other teas.

P58 Multi-Residue Pesticide Analysis in Herbal Teas Using the QuEChERS Extraction, Cartridge Solid Phase Extraction Cleanup and Comprehensive Two-Dimensional Gas Chromatography Time-of-Flight Mass Spectrometry

Michelle Misselwitz, Jack Cochran, and Julie Kowalski
Restek Corporation, 110 Benner Circle; Bellefonte, PA 16823, USA; michelle.misselwitz@restek.com

Herbal tea, a non-caffeinated drink made from plants, herbs, or spices has been used throughout history for its potential medicinal benefit. Used frequently in Traditional Chinese Medicine (TCM) different blends of herbal material will be formulated depending on the desired medicinal properties. As with any plant based commodity, there is the potential for pesticide residues to remain in the final product.

Dried plant material found in herbal tea poses a significant challenge to the analytical chemist to detect trace levels of pesticide residues. The extract, even after an extensive cleanup can contain a large amount of coextractive material that can completely overwhelm the target pesticides, making trace detection very difficult. Furthermore, nonvolatile material not removed during extract cleanup deposit onto the inlet and column requiring more frequent maintenance to be performed.

We employed the QuEChERS methodology for a quick extraction of store bought herbal teas and a combination solid phase extraction (cSPE) cartridge cleanup containing 500mg carbon and 500mg primary secondary amine (PSA). The percent recoveries of spiked pesticide standards and quantification of incurred pesticides were determined using comprehensive two-dimensional gas chromatography time-of-flight mass spectrometry (GCxGC-TOFMS).

P59 Does the QuEChERS Solvent Acetonitrile Damage GC Columns?

Jack Cochran, Michelle Misselwitz, Julie Kowalski, and Chris Rattray
[1]**Restek Corporation**, 110 Benner Circle, PA 16823, USA; jack.cochran@restek.com

Claims of GC column damage from acetonitrile used for QuEChERS extractions have been reported in presentations and scientific literature, but no definitive experimental results prove this to be true. We performed studies on 30m x 0.25mm x 0.25µm 5% phenyl-type GC columns, the format most used for multi-residue pesticide GC analyses, to define any performance degradation for pesticide retention time stability, pesticide response factor, and GC column stationary phase bleed that might result from using either acetonitrile or acetonitrile with 1% acetic acid, the two QuEChERS solvents.

P60 Same Separation with Half the Column: Extending the Lifetime of your GC Column with Column Trimming Maintenance and Method Translation

Jack Cochran, Michelle Misselwitz, and Jason Thomas
Restek Corporation, 110 Benner Circle; Bellefonte, PA 16823, USA; jack.cochran@restek.com

Polybrominated diphenyl ethers (PBDEs) have been found to be persistent and bioaccumulative in the environment. The technical mixtures containing penta and octa congeners were voluntarily withdrawn in the United States in 2005 and the last remaining PBDE mixture, decaBDE, should be completely phased out by the end of 2013. While these mixtures have been phased out of production and use, the concentrations in the environment have not been declining and are currently still widely monitored.

The analysis of PBDEs is challenging due to structural isomers that need to be chromatographically separated and thermally label compounds of interest that may breakdown during gas chromatography. PBDEs included in EPA Method 1614 are well resolved on a 15m x 0.25mm x 0.10µm Rtx-1614 GC column, a 5% diphenyl, 95% dimethyl polysiloxane type phase that was specifically designed to meet method resolution requirements. Using a short, thin film column also allows the elution of decabromodiphenyl ether (BDE-209) without on-column thermal degradation.

Monitoring efforts of the levels of PBDEs include a wide array of biota and environmental matrices. Non-volatile material may still persist even in cleaned-up final extracts, requiring GC column and inlet maintenance to be performed. Using a 15m x 0.25mm x 0.10µm column, how many loops of the GC column can one clip for maintenance before the Method 1614 resolution requirements of BDE 49 and BDE 71 can no longer be met? The resolution between BDE 49 and 71 must be less than 40% valley height to meet method criteria.

P61 Analysis of Regulated Mycotoxins in Infant Formula using Liquid Chromatography-Tandem Mass Spectrometry

Dan Hengst and Katerina Mastovska
Covance Laboratories, 3301 Kinsman Boulevard; Madison, WI 53704-2523, USA; Dan.Hengst@covance.com

The analysis of mycotoxins in infant formulas is challenging due to the very low limits of quantitation required to comply with world-wide regulations. For example, the EU regulated limits (EC Regulation 1881/2006) for aflatoxin M_1, aflatoxin B_1, and ochratoxin A are 0.025, 0.1, and 0.5 µg/kg, respectively. Most current and traditional methods require labor-intensive and time-consuming sample purification and concentration steps to achieve these levels using liquid chromatography with fluorescence detection or liquid chromatography-mass spectrometry. In this study, an Agilent 1290 UHPLC coupled to an Agilent 6490 triple quadrupole mass spectrometer was utilized to analyze the EU regulated mycotoxins (aflatoxins B_1, B_2, G_1, G_2, M_1 and non-regulated M_2; ochratoxin A; deoxynivalenol; zearalenone; fumonisins B_1 and B_2; and T-2 and HT-2 toxins) in infant formula using a simple extraction without any clean-up and/or concentration step. The triggered MRM (tMRM) function of the system was employed for increased confidence in positive analyte identification even at the very low, sub-ppb concentration levels in the complex infant formula matrix.

P62 Simultaneous Determination of 13 Mycotoxins in Rice by LC/MS/MS

Dong-Ho Kim, Hyun-Jeong Jo, Jae-Min An, Hye-Jeong Yun, Sung-yeun Kim, Byeong-Cheol Son, Dong-Keun Jo, and Byung-Lim Cho
National Agricultural Products Quality Management Service, 80, Seonyudong 1-ro, Youngdeungpo-gu, Seoul, Korea; anoldmu@korea.kr

Thirteen mycotoxins, including aflatoxins(B_1, B_2, G_1, G_2), ochratoxin A, fumonisins(B_1, B_2), zearalenone, T-2 toxin, HT-2 toxin, nivalenol, deoxynivalenol, and 3-acetyldeoxynivalenol were analyzed simultaneously in the rice produced in Korea by liquid chromatography coupled with triple quadrupole mass spectrometry (LC/MS/MS). All samples were extracted with 70% methanol and cleaned up by immunoaffinity column containing various antibodies, at one time. 20% acetonitrile containing 2mM ammonium acetate and acetonitrile containing 0.3% formic acid were used as the mobile phases and the C_{18} column was applied for the separation of 13 mycotoxins. Limits of quantification (LOQ) were 0.05 µg/kg for aflatoxins (B_1, B_2, G_1, G_2), and T-2 toxin, 0.1 µg/kg for HT-2 toxin, zearalenone, and ochratoxin A, and 1.0 µg/kg for nivalenol, deoxynivalenol, 3-acetyldeoxynivalenol, and fumonisins (B_1, B_2). Recoveries for 13 mycotoxins ranged from 70 to 109% with RSD < 20%. Nivalenol, deoxynivalenol, fumonisins, and zearalenone were the mycotoxins mainly detected in the tested rice samples.

P63 Using the Power of Comprehensive Two-Dimensional Gas Chromatography Time-of-Flight Mass Spectrometry for the Non-Target Screening of Natural Products in Herbal Tea

Michelle Misselwitz, Jack Cochran, and Julie Kowalski
Restek Corporation, 110 Benner Circle; Bellefonte, PA 16823, USA; michelle.misselwitz@restek.com

Comprehensive two-dimensional gas chromatography time-of-flight mass spectrometry (GCxGC-TOFMS) provides enhanced separation and increased peak capacity compared to a one-dimensional GC analysis. The extra peak capacity and resolution is beneficial for separating potentially important biologically active analytes in complex herbal tea extracts that would otherwise be masked in matrix interferences in a one-dimensional GC analysis.

Herbal tea, a non-caffeinated drink made from plants, herbs, or spices has been used throughout history for its potential medicinal benefit. Used frequently in Traditional Chinese Medicine (TCM) different blends of herbal material will be formulated depending on the desired medicinal properties. A South African tea, Sceletium & Honeybush Tea, contains active alkaloids that have been isolated and formulated into a pill format. The active alkaloid, mesmebrine, has been found to reduce stress, increase energy and uplift your mood. Other herbal teas contain many natural products that could be beneficial biologically active compounds.

Herbal tea samples were extracted using the QuEChERS method. A split injection of the tea extract with no cleanup was used to screen the samples for natural products. The use of GCxGC-TOFMS was very beneficial for data mining of non-target analytes in the herbal tea extracts. We took advantage of the full mass spectra for NIST library searches, and the ordered chromatograms produced by GCxGC.

P64 Enhanced Degradation of Organochlorine, Organophosphorus, Organonitrogen, and Carbamate Pesticides during Hot GC Splitless Injection of QuEChERS Extracts of Canola Seed

Julie Kowalski, Michelle Misselwitz, and Jack Cochran
Restek Corporation, 110 Benner Circle, Bellefonte, PA 16823, USA; julie.kowalski@restek.com

We noticed low response factors for select pesticides with hot splitless injection GC of EN QuEChERS extracts of canola seeds compared to those same pesticides in EN QuEChERS extracts of tobacco. An experiment was designed to quantify the differences by GC analyzing pesticide-spiked tobacco and canola extracts, and acetonitrile solvent-only standards. The pesticide concentrations were relatively high at 5 ng/µL to minimize any matrix-enhanced response effect, especially for the solvent-only standards. A 4mm ID single taper with wool liner was used for the work at 250°C.

Organochlorine pesticides that showed markedly low response factors when in canola seed extracts included chlorothalonil, delta-BHC, and endosulfan sulfate. Interestingly, alpha- and gamma-BHCs, and endosulfans I and II were not impacted. Other pesticides that had low responses in canola extracts versus tobacco extracts and solvent-only standards were carbaryl, methiocarb, dichlofluanid, captan, folpet, deltamethrin, and more. LC-MS/MS analysis of canola and tobacco extracts, and solvent-only standards, gave essentially the same response factors for all pesticides independent of the matrix, which proves that the low GC response factors were due to the canola extract. One theory is that isothiocyanates in the extracts from canola are leading to pesticide degradation during hot splitless injection. It is possible too that this effect is enhanced by having a wool-packed GC inlet liner.

P65 Simple Acetonitrile Extraction of Quaternary Ammonium Compounds (QAC) from Fruit and Vegetables and Quantitative Analysis by UPLC-MSMS and UPLC-QTOF-MS

Laura M. Melton, K.J. Viezens, A. Giela, M.J. Taylor, and K.B. Reid
Science and Advice for Scottish Agriculture (SASA), a division of the Scottish Government, Roddinglaw Road, Edinburgh, EH12 9FJ, UK; laura.melton@sasa.gsi.gov.uk

Benzalkonium chloride (BAC) and didecyl dimethyl ammonium chloride (DDAC) are quaternary ammonium compounds (QAC) used for both disinfectant and plant protection purposes. Recently in Europe unexpected residues of QAC above the current statutory maximum residue level (MRL) of 0.01 mg/kg have been found in foodstuffs. A higher non-statutory MRL of 0.5 mg/kg has been assessed for potential toxicological effects and judged to be safe. This MRL has been adopted to allow marketing of produce with residues of QAC above the statutory MRL. European countries are required to carry out monitoring on residues of BAC and DDAC in food and feed to allow a substantive statutory MRL to be set.

SASA (official lab) participates in the annual UK and coordinated EU pesticide residues in food surveillance programmes on behalf of the Scottish Government. A simple (multi-residue) acetonitrile extraction that does not require any solvent evaporation, solvent exchange or sample clean-up has been validated for over 40 pesticides in a range of fruits and vegetables. It was relatively straightforward to incorporate BAC with chain lengths of n=10, 12, 14 and 16 and DDAC into this acetonitrile multi-residue method. The quantitative determination of these analytes has been successfully validated in aubergine, cherries, green beans, leeks, peas and strawberries.

A common acetonitrile extract can be analysed both by UPLC-MSMS and UPLC-QTOF-MS providing simultaneous screen and confirmation results. The method has been used successfully to quantify residues of DDAC in leek and in a recent European Union proficiency test on potato homogenate.

P66 A Novel HILIC Column for the Determination of Paraquat and Diquat in Environmental Samples

Jeremy C. Shia, Kim Van Tran, and Michael S. Young
Waters Corporation, 5 Technology Drive, Milford, MA 01757, USA; michael_s_young@waters.com

Paraquat and diquat (quats), doubly charged quaternary ammonium compounds, are herbicides that are extensively used worldwide. Ion-pair reversed-phase LC-UV is commonly used for analysis of these compounds in commodities and drinking water. However, the ion-pairing reagents used are not usually suitable for LC-MS analysis. Recently we have reported highly sensitive LC-MS methods for the quats. These methods use HILIC, an alternative type of LC separation for polar compounds. The HILIC mode is a better choice for LC-MS analysis because no ion-pairing reagents are needed. However, HILIC separation for UV detection has not been adopted because baseline separation of paraquat and diquat has been difficult to achieve. Recently, we reported a new UltraPerformance LC® column for HILIC chromatography. Baseline separation of the two quat compounds is easily achieved with this new column so it can be used with either UV or MS detection. In this study, the new HILIC column is demonstrated for the analysis of diquat and paraquat in drinking water. Extraction and clean-up was accomplished using a mixed mode weak cation exchange SPE cartridge (Oasis WCX) prior to LC analysis. The separation achieved on the new HILIC column enables efficient and sensitive methods for the determination of quats in drinking water LC-MS or LC-UV. LOQs below 50 ng/L ppb was achieved using LC-MS and below 500 ng/L using LC-UV.

P67 **A Novel Approach to the Reduction of False Positive and Negative Identifications in Screening of Pesticide Residues in Food Analysis**

Severine Goscinny[1], Michael McCullagh[2], Kieran Neeson[2], Jeff Goshawk[2], David Eatough[2], Sara Stead[2], Ramesh Rao[2], Dominique Roberts[2], and Kenneth Rosnack[3]
[1]Wetenschappelijk Instituut Volksgezondheid Institut Scientifique de Santé Publique, Rue Juliette Wytsmanstraat 14, 1050 Brussels Belgium
[2]Waters Corporation, Floats Road, Wythenshawe, Manchester, UK
[3]**Waters Corporation**, 34 Maple St., Milford, MA, USA; ken_rosnack@waters.com

Current trends indicate that more than 500 compounds are routinely used under strict regulation on a global basis. With increasing global trade there is a requirement for multi-analyte screening strategies capable of efficiently detecting residue violations to protect consumer safety. Benefits of full spectra acquisition and the specificity of accurate mass measurement is well characterized and is used in combination with, time tolerances, isotope fits, fragment ions/ratios and response thresholds to reduce false positive/ negative identifications in screening assays. Nonetheless, it is a challenge to identify targeted compounds present in the sample with a large number of co-extracted matrix components. The application of ion mobility to remove false positive identifications and importantly false negative identifications will be presented. The assay is based on the analysis of sample extracts and matrix matched calibrants of pear, ginger, leek and mandarin, as well as quality control samples generated for an EU-RL proficiency test. UPLC HDMS^E drift times generated from the solvent standards and the matrix matched calibrants were shown to statistically belong to the same population. Hence it can be shown that the drift time of the residues is independent of the matrix and can be utilized as a confirmatory parameter to increase confidence in identification. The drift time data generated was entered into a scientific library within a new scientific information system. This allowed the expected and determined drift times to be utilized to reduce false identifications in the proficiency test samples and matrix matched calibrant series analyzed.

P68 **Application of Micro Fluidics MS for the Screening of Pesticide Residues in Food Analyses**

Severine Goscinny[1],David Douce[2], Michael McCullagh[2], Dominique Roberts[2], Sara Stead[2], and Kenneth Rosnack[3]
[1]Wetenschappelijk Instituut Volksgezondheid Institut Scientifique de Santé Publique, Rue Juliette Wytsmanstraat 14, 1050 Brussels Belgium
[2]Waters Corporation, Floats Road, Wythenshawe, Manchester, UK
[3]**Waters Corporation**, 34 Maple St., Milford, MA, USA; ken_rosnack@waters.com

Full spectra acquisition and the specificity of accurate mass measurement is well characterized and used in combination with time tolerances, isotopic match, fragment ions/ratios and response thresholds to reduce false positive or 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. Dilution of matrix to reduce “matrix effects” is possible but limited by the detection threshold for the targeted compounds. Micro fluidic UPLC has been explored to determine the applicability of screening for pesticide residues using this technology. The assay is based on the analysis of sample extracts, matrix matched calibrants (pear, ginger, leek and mandarin) and quality control samples generated for an EU-RL proficiency test (FV-13, FV-14, SM3 and SM4). These samples were analyzed using the micro fluidic LCMS interface. The source incorporates a ceramic micro fluidic device that contains an analytical column (150µm x 50mm) along with the ionization emitter in one interchangeable device.

Micro fluidic UPLC MS^E was first acquired for a series of solvent standard mixtures and results were utilized to generate retention time information within the pesticide library of the scientific information system. Initial results have shown gains in both sensitivity and signal to noise with no compromise in the linearity correlation coefficients for the matrix matched calibrants (r2≥0.99).

P69 Analysis of Multiple Pesticides by Supercritical Fluid Chromatography/Tandem Mass Spectrometry with Sub-2 Micron Particle Column - A Feasibility Study

Jinchuan Yang, Brian Tyler, Joseph P. Romano, and Jennifer A. Burgess
Waters Corporation, 34 Maple St, Milford, MA 01757, USA; joe_romano@waters.com

GC-MS and LC-MS are common techniques for pesticide residue analysis and both are required due to the wide range of physiochemical properties of pesticides. It has been reported that SFC can be used as a single separation technique for the simultaneous analysis of LC-amenable and GC-amenable pesticides. This unique SFC approach provides a simple and convenient solution for multiple pesticide residue analysis. With the new development of UltraPerformance Convergence Chromatography (UPC^2), its applicability and potential advantage in multiple pesticide residue analysis has been investigated. UPC^2 is a separation technique that uses compressed carbon dioxide as the primary mobile phase. It takes advantage of the unique physical properties of compressed carbon dioxide (at or near supercritical state), sub-two micron particle chromatography columns and an advanced chromatography system design to achieve unique selectivity, high efficiencies and speed. In this work, we present a feasibility study using UPC^2 with ultra sensitive tandem quadrupole MS/MS detection for 18 pesticides with a wide range of polarities (logPow=-4.6 to 7.1) and molecular weights (112-889). This study focused on UPC^2 separation, interfacing with MS, and MS/MS detection. The sensitivity and repeatability of the multi-pesticide residue analysis in the presence of common food matrices, such as spinach, wheat flour, and apple juice will be presented.

P70 Validation of an Accurate Mass Screening Method for Pesticide Residues in Food using UPLC-QToF MS and Automated Data Processing Software

Sara Stead[1], Dominic Roberts[1], Jennifer A. Burgess[2], Ramesh Rao[1], Monica Lopez Garcia[3], and Richard Fussell[3]
[1]Waters Corporation, Manchester, UK;
[2]**Waters Corporation**, 34 Maple St, Milford, MA 01757, USA; jennifer_burgess@waters.com;
[3]Food and Environment Research Agency, York, UK

Regulatory authorities face increasing pressure to ensure the safety of the global food supply. Pesticides are high on the list of consumer concerns and laboratories are tasked to screen samples for multiple residues in a single analysis within an appropriate timescale and cost. The approaches currently being developed are based on the use of High-Resolution Mass Spectrometry (HR-MS) instrumentation with non-targeted acquisition operated under generic conditions, followed by targeted processing against a database of typically several hundreds of pesticides. The key to the successful implementation for routine analysis will be the capability of the system to accurately detect residues at low concentrations with an acceptable level of false positive and false negative detects, as outlined in the EU guidelines (SANCO/12495/2011).

In this study, we report the validation of a screening method using UPLC coupled to QToF-MS and automated data processing software for the determination of approximately 200 pesticides at a screening detection limit (SDL) of 0.01 mg kg^{-1} in a range of representative matrices. QToF technology has been used to provide information rich data with full spectra acquisition and excellent sensitivity. Structural elucidation information for all the identified pesticides was generated by simultaneous acquisition of accurate mass precursor and accurate mass fragment ions. Using this approach a high overall detection rate with a low number of false negative and false positive detects at the EU regulatory limit of 0.01 mg kg-1 was achieved, demonstrating the applicability of this solution for routine screening analysis in wide variety of matrices.

P71 Next Generation Pesticide Analysis Using Microfluidic Nano-LC-MS/MS

Gregory T. Roman, Gareth Cleland, Jim Murphy, Jennifer A. Burgess, and Geoff Gerhardt
Waters Corporation, 34 Maple St, Milford, MA 01757, USA; gregory_roman@waters.com

Recent advances in MS and MS/MS technologies have enabled the widespread application of these technologies for multi-residue trace level contaminant analysis in residue testing laboratories. Challenges, however, continue with increasingly stringent legislation and the sample preparation required to address matrix complexity. The ability to further decrease the levels of detection obtainable with MS/MS systems is still of high importance in order to improve detection of challenging analytes, increase the precision and accuracy of low level detections and reduce matrix effects through dilution or reduced sample loading. Here we demonstrate the implementation of a prototype microfluidic device coupled with tandem mass spectrometry for the rapid screening of pesticides in example food matrices (infant formula, summer squash, and ginger root). Microfluidic nano-LC compared to conventional UPLC shows significant improvements in sensitivity. The sensitivity improvement for the microfluidic device can be attributed to improved ionization efficiency at low flows (2.3 uL/min) and a reduction in the dilution effect present in 150 µm diameter channels compared to a 2.1 mm column. Microfluidic devices were challenged with 300 replicate infant formula matrix injections. Pressure profiles were very similar with an increase in pressure of only 1.3%. Peak area reproducibility for a set of 10 pesticides demonstrated an average of 7.5 %RSD, and retention time reproducibility of < 1.0% (n = 300 injections). An additional advantage with microfluidic chromatography is the reduction in solvent consumption. With the application presented here, the microfluidic method used 0.6%

of the total volume of solvent required for the UPLC method. Over the course of a year this has the potential to result in major cost savings to laboratories.

P72 A Sensitive UPLC/MS/MS Method for Determination of ACQ Wood Preservative Quat in Environmental Waters

David M. Mauro[1] and Michael S. Young[2]
[1]META Environmental, Inc., 49 Clarendon Street, Watertown MA 02472 USA;dmauro@metaenv.com
[2]**Waters Corporation**, 5 Technology Drive, Milford, MA 01757, USA; michael_s_young@waters.com

Wooden utility poles must be treated with preservatives to protect against insects, fungi, and other environmental damage. A number of different preservatives have been employed for this purpose such as pentachlorophenol, coal tar creosote and copper chromium arsenic, but the toxicity of these substances is of environmental concern. The Electric Power Research Institute (EPRI) is investigating the effectiveness of commercially available prevention methods to reduce preservative migration from treated wood poles and to compare the migration of constituents of various wood treatments.

Ammoniacal copper quat (ACQ) is one of the treatment chemicals being studied. ACQ combines copper oxide with the quaternary ammonium ion DDA+ (didecyldimethylammonium). Over the past eight years a number of wooded poles, treated with ACQ, were installed at a study site in Florida. Each pole was planted above a custom made lysimeter designed to gather rainwater that contacts the poles and percolates through the ground. Periodically, water was collected from the lysimeters to be analyzed for DDA+. A highly sensitive and selective method was developed for determination of DDA+ in the water samples taken from the lysimeters. Solid-phase extraction (SPE) was performed using Oasis WCX, a mixed-mode weak cation-exchange SPE sorbent. After SPE, the isolated DDA+ was quantified using UPLC/MS(MS). Detection limits well below 1 µg/L (ppb) were demonstrated for groundwater or surface water.

P73 Detection of Underivatized Glyphosate and Similar Polar Pesticides in Food of Plant Origin by LC-MS/MS

Stephen Lock[1], Herrmann Unterluggauer[2], Farzad Pakdel[3], and André Schreiber[3]
[1]**AB SCIEX**, Phoenix House, Lakeside Drive, Warrington, UK; stephen.lock@absciex.com;
[2]Austrian Agency for Health and Food Safety (AGES), Innsbruck, Austria;
[3]AB SCIEX, Concord, ON, Canada

Glyphosate is a common broad-spectrum systemic herbicide used widely to kill weeds especially annual broadleaf weeds and grasses known to compete with crops. Usually Glyphosate, as it is very polar, undergoes FMOC derivatization by reacting the native glyphosate with fluorenylmethyloxycarbonyl chloride (FMOC-Cl) before analysis. This derivatization step complicates the analysis and there is a growing need for a method which can detect not only Glyphosate (and its major metabolite AMPA) but also Glufosinate and similar highly polar compounds, in their underivatized states. In addition a simplified approach to sample extraction using either QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) or a solvent extraction would be beneficial. Here we present initial data using a new LC-MS/MS method which combines the use of a HILIC type chromatography on an AB SCIEX QTRAP 5500 system to detect underivatized Glyphosate; AMPA, Glufosinate, and Ethephon which have been spiked in different food matrices. A simple solvent extraction using the QuPPe protocol has been used. All the compounds were identified and quantified using two MRM transitions at 0.1 mg/kg after 5x dilution of QuPPe extracts. However, matrix effects were observed so in routine analysis it is recommended that matrix matched calibration standards or ideally heavy labeled internal standards are used.

P74 Automated Derivatization, Cleanup and LC-MS/MS Determination of Glyphosate and Others Polar Pesticides

André Schreiber[1] and Oscar Cabrices[2]
[1]**AB SCIEX**, 71 Four Valley Drive, Concord, ON, Canada; andre.schreiber@absciex.com;
[2]Gerstel, Baltimore, MD, USA

Glyphosate is a common broad-spectrum systemic herbicide used widely to kill weeds especially annual broadleaf weeds and grasses known to compete with crops. There is an interest in the reliable and sensitive quantitation and identification of glyphosate residues, its metabolite AMPA, and the related glufosinate in food and water.
Commonly large volume injection into ion chromatography or LC systems based on HILIC followed by sensitivity MS/MS detection is used for analysis. However, interference can influence results in complex samples since the method does not use any cleanup. Derivatization techniques can be used successfully. The method presented here uses derivatization with FMOC-Cl followed by automated SPE cleanup using a Gerstel front-end and detection using LC-MS/MS with an AB SCIEX QTRAP® 4500 system. Limits of quantitation in food were found below the target 100 µg/kg allowing dilution to minimize potential ion suppression. In drinking water samples glyphosate was quantified below 0.1 µg/L. Linearity of over three orders of magnitude with $r > 0.999$ was observed with excellent reproducibility because of the complete automation of the sample handling procedure.

P75 A Sensitive Multi-matrix Analysis of Glyphosate and Related Compounds by HPLC/MS/MS

Serena Lazzaro[1], Sky Countryman [1], Dirk Hansen [2], Robert Brockmeyer [3], and Allen Misa[1]
[1] **Phenomenex,** 411 Madrid Avenue, Torrance, CA 90501, USA; SerenaL@phenomenex.com
[2] Phenomenex, Aschaffenburg, Germany; [3] Eurofins Sofia GmbH, Berlin, Germany

Glyphosate is one of the most widely used herbicides. During recent years there has been an increasing demand for reliable, selective and sensitive methods for residue analysesalso because of the diversity in the MRLs of glyphosate and related compounds (AMPA and Glufosinate) on trade commodities across the different nations acting as a trade barrier.

A simple and robust analysis method has been developed. Although requiring a sensitive instrument, this approach has the advance of almost completely avoiding matrix effects. The simplified sample preparation procedure provides a simple acidic extraction followed by derivatization with 9-fluorenylmethylchroroformate (FMOC-Cl) without having the necessity to a further cleaning up. After pH adjustment to stop the derivatization reaction the FMOC derivatives are analysed without any further cleanup using an HPLC-MS/MS system equipped with a Phenomenex Gemini 3µm C18 column and ABSciex API5000 tandem mass spectrometer. The method has been in-house validated by the Eurofins Sofia laboratory. It achieves a limit of quantification (LOQ) of 0.01 mg/kg for all analytes and in four different matrices. The procedure, originally developed for water samples, was adapted by using a strongly diluted hydrochloric acid extracts of lentils, oil seeds, wheat and tea with recovery values within the range 81- 101%.

P76 A Fast, Selective and Sensitive LC/MS/MS Analysis of Chloramphenicol in Shrimp

Serena Lazzaro, Matthew Trass, Sky Countryman, and Allen Misa
Phenomenex, 411 Madrid Avenue, Torrance, CA 90501, USA; SerenaL@phenomenex.com

Chloramphenicol (CAP) is a broad-spectrum antibiotic with historical veterinary uses in all major food-producing animals, humans and companion animals. In 2001 and 2002, chloramphenicol residues were identified in seafood imported to EU, Canada and the U.S. from China. More sensitive methods were needed to control and monitor these residues in imported food, particularly in shrimp. The EU, with the intend to harmonize the analytical methods performance for substances for which no MRL has been established, set 0.3 ppb as minimum required performance limit (MRPL), the minimum content of CAP which at least has to be detected and confirmed. The USFDA method for CAP in shrimp has 0.08 ppb as limit of detection (LOD) and 0.3 ppb as limit of quantitation (LOQ). The use of Strata-X solid phase extraction (SPE) cartridges for cleanup and sample concentration and the ultra-fast Kinetex C18 core-shell technology allow to achieve the selectivity and sensitivity necessary to quantify CAP in less than 5 minutes and with a LOQ of 0.01 ppb. This method has proven to be rugged, reliable, and reproducible across several classes of matrices at a variety of sample levels.

P77 Levels of Copper in Hop Samples Used for Brewing

Mehmet Coelhan
Technische Universität München, Research Center Weihenstephan for Brewing and Food Quality, Alte Akademie 3, 85354 Freising, Germany; coelhan@wzw.tum.de

Several varieties of female hops (*Humulus lupulus L.)* are used in brewing process primarily for two purposes; a) for bittering and b) for hoppy flavor, while in early years extending of shelf life of beer by hope may also have played a role. The economic value of the hop plant is derived from its worldwide application as an essential flavoring ingredient in the brewing industry. Downy mildew (*Pseudoperonospora humuli* L.) is a serious fungal disease which threats hop cultivation in many hop-growing areas of the world. The killing of stems, blossoms, and cones greatly reduces the quality and quantity of the crop. As first symptoms of downy mildew, spike like infected growths arise from the crown among normal, slender shoots, which are usually pale green or silvery gray in color, rigid stocky and stunted. Before the spores appear, infected leaf stalks and other infected parts of the vine often present a grayish blistered appearance. The biggest economic losses from powdery mildew infection are the cost of control and reduction in yield and quality due to cone infection. Even low infection levels, particularly in aroma hop varieties, can result in a loss of hop quality. In some cases, particularly for organic hop cultivation, only copper containing formulations may be used for protection. In the present study levels of copper were investigated in more than 200 hop samples. It was found that copper concentrations vary in a wide range. Some samples contained copper at higher levels than 1000 ppm.

P78 A Selective Method to Identify and Quantify Mycotoxins in Cereal Based Food: Simple Extraction with Analysis by Kinetex® Core-Shell Technology and Tandem Mass Spectrometry with Fast Polarity Switching

Serena Lazzaro[1], Sky Countryman[1], Andre Schreiber[2], and Jianru Stahl-Zeng[3]
[1]**Phenomenex**, 411 Madrid Avenue, Torrance, CA 90501, USA; SerenaL@phenomenex.com
[2] ABSCIEX , ON, Canada; [3] ABSCIEX, Germany

Mycotoxins contamination of food and agricultural commodities poses a risk to health due to their toxic effects. To protect consumers, many countries set maximum limits (MLs) with widely varying tolerances among different national or multilateral agencies. Various factors play a role in MLs' decision-making process. These include economic and scientific factors, to assess risk, and analytical methodology. In the recent decades Liquid Chromatography coupled with tandem Mass Spectrometry (LC-MS/MS) has revolutionized the analysis of mycotoxins. One of major advantages of LC-MS/MS, with respect to well-established UV or fluorescence detection methods, is the possibility to analyze several classes simultaneously. However, to ensure unambiguous identification and accurate quantification a selective chromatographic method is indispensable to resolve analytes from matrix interferences.
A rapid, robust, sensitive "Dilute-and-Shoot" LC-MS/MS method has been developed to quantify several classes of mycotoxins in cereal based foods. Kinetex core-shell HPLC columns and a triple quadrupole MS deliver a fast, highly selective and sensitive method

minimizing the potential risk of false positive and negative results. The method, tested on several cereal based samples, permits to quantify in only 8 minutes seventeen toxins with a Limit of Quantification (LOQ) below the stringent MLs required by Commission Regulation (EC) N 1881/2006.

P79 Solid Phase Extraction and Core-Shell Technology for Rapid Detection, Identification and Quantification of Phenylbutazone in Ground Meat

Serena Lazzaro, Seyed Sadjadi, Sky Countryman, and Terrell Mathews
Phenomenex, 411 Madrid Avenue, Torrance, CA 90501, USA; SerenaL@phenomenex.com

The European community was outraged when it discovered horsemeat had been illegally incorporated into food products. As horses can be classified as food animals, equal concern was raised for the potential of veterinary drugs, banned for human consumption, entering the food supply. For human safety, the European Commission has set maximum residue levels for veterinary drugs in food of animal origin. Phenylbutazone is a non-steroidal anti-inflammatory drug commonly prescribed for horses. Due to its toxicity, phenylbutazone is prohibited for human use. The recommended concentration for phenylbutazone, as proposed by the EU reference laboratory is 5ppb.

Testing for phenylbutazone is a challenge due impart to the complex sample matrix requiring selective extraction/cleanup to prevent false positive results by LC/MS/MS analysis. We present a simple and robust method for extraction and cleanup of phenylbutazone from meat, using Strata X-A SPE cartridge with recovery values of 108.2%, 100.7% and 90.3% in matrix-matched spiked sample at 2, 5 and 75 ppb. This highly selective HPLC method using Kinetex 2.6 µm XB-C18 core-shell technology enabled LC/MS/MS run times in less than 5 minutes, maximizing sample through put. The method provides a limit of detection (LOD) of 1 ppb and a limit of quantitation (LOQ) of 2 ppb; well below the recommended concentration of 5ppb proposed by the EU reference laboratory for NSAIDs.

P80 The Detection of Trenbolone and Melengestrol in Meat Samples by LC-MS/MS

Fabrice Monteau[1], Stephen Lock[2], Loic Beyet[3], Lauryn Bailey[4], and Bruno Le Bizec[4]
[4]**AB SCIEX**, 500 Old Connecticut Path, Framingham, MA, USA; lauryn.bailey@absciex.com
[1] LABERCA, Nantes, France; [2] AB SCIEX, Warrington, UK; [3] AB SCIEX, Paris, France

Trenbolone and Melengestrol are steroids used by veterinarians on livestock to increase muscle growth and appetite. Melengestrol is approved for use as a growth promoter in livestock, including beef cattle, in the United States but both are not approved in the European Union and were prohibited in 1988. These steroids normally exist at low levels in meat imported into the EU and therefore low limits of detection are required. Although both Trenbolone and Melengestrol are normally administered to cattle in the ester form e.g. acetates, they are quickly metabolized to the native steroids. This work shows where LC-MS/MS can be used to detect Trenbolone and Melengestrol at low levels in real samples.

To improve the sensitivity of the assay both compounds are acetylated before samples were extracted. The acetate derivatives were then analyzed by reverse phase high performance liquid chromatography with electrospray mass spectrometry. Both MRM and MRM^3 methods were developed by infusion of the acetate standards and the sensitivity of both methods were compared by analyzing meat extracts.
MRM quantitation has shown that both steroids can be detected at low part per trillion levels with some of the interference peaks removed by the use of MRM^3 instead on MRM methods.

P81 Cleanup Protocols for Multi-Residue Pesticide Analysis of Dried Teas

Kim Van Tran and Michael S. Young
Waters Corporation, 5 Technology Drive, Milford, MA 01757, USA; michael_s_young@waters.com

The QuEChERS methods have simplified and streamlined sample preparation for pesticide analysis of many fruits and vegetables. Although effective for many types of samples, there are challenges when this technique is applied to certain dried commodities such as teas. Not only must the proper amount of water be added and equilibrated prior to QuEChERS extraction, but highly resinous leafy materials such as tea may require significant cleanup prior to LC-MS and GC-MS analysis. This poster will present optimized strategies for multi-residue pesticide analysis of teas. After optimized QuEChERS extraction, three aliquots are taken for further analysis. One aliquot is taken for determination of acidic compounds not amenable to cleanup on PSA. A second aliquot is taken for determination of LC-MS analyzable compounds and is subjected to a dSPE cleanup specific for that analysis. The third aliquot is subjected to an SPE cartridge cleanup optimized for GC-MS analysis. This poster will discuss the cleanup protocols chosen for each group of compounds (LC-MS acids, LC-MS base/neutrals and GC-MS) and present method performance data for target pesticides extracted from green and black tea.

P82 Rapid and Sensitive analysis of Eight Artificial Sweeteners in Environmental Waters by Direct Injection UPLC-MS/MS

Dominic P. T. Roberts[1], Kendon S. Graham[2], and Michael J. Scotter[3]
[1] Waters MS Technologies Centre, Manchester, UK, [2]**Waters Corporation**, Milford, MA 01757, USA; kendon_graham@waters.com
[3]The Food and Environment Research Agency, Sand Hutton, York, UK

Artificial sweeteners are permitted for use as sugar substitutes in foods, animal feeds, beverages, and sanitary products worldwide. Their advantage to the food industry is that once ingested they provide negligible calorie intake or glycemic effect in the body. Some of these compounds are not readily metabolised by mammals allowing them to be excreted unchanged in urine and faeces. After decades of use, their occurrence is of particular concern since they are known to pass through wastewater treatment plants unchanged, allowing them to bio-accumulate within the environment. Hence, there is a requirement for a sensitive multi-analyte quantitative method to be able to detect this new group of emerging contaminants in a range of environmental matrices at levels much lower than those used for sweetening foods and beverages. Here we present a sensitive and robust method for the determination of 8 sweeteners in environmental waters by direct injection. Separation of the sweeteners was achieved using a Waters BEH Phenyl 100 x 2.1mm, 1.7µm column and detection was performed on a Waters Xevo TQ-S UPLC-MS/MS system using electrospray ionisation. The enhanced sensitivity of the Xevo TQ-S allowed for the direct injection of 10 µl water. The method was validated in house and showed excellent limits of detection ranging from 0.5 to 10 ng/L and linearity R^2>0.991. Precision in matrix ranged from 1.9 to 5.8 RSD%. Matrix suppression was minimal ranging from 78 to 111%. Acesulfame-K and sucralose were detected in a river water sample at 26 and 45 ng/L, respectively.

P83 Determination of Pesticide Residue in Apple Juice Using the AutoMate-Q40

Tyler Trent[1], Rick Jordan[2], and Tom Hartlein[1]
[1]**Teledyne Tekmar**, 4736 Socialville Foster Road; Mason, Ohio 45040, USA; Tyler.Trent@teledyne.com
[2]Pacific Agricultural Laboratory, 12505 N.W Cornell Road; Portland, Oregon 97229, USA

QuEChERS is a Quick-Easy-Cheap-Effective-Rugged-Safe extraction method that has been developed for the determination of pesticide residues in agricultural commodities. While the original unbuffered method was developed for plant matrices, since 2003, two additional buffered methods were created and adapted to many additional matrices such as fruit juices. The rise in popularity of the QuEChERS technique and the increase in sample testing have driven the need for automation for this extraction technique. The AutoMate-Q40 streamlines the two part QuEChERS method from the liquid extraction to the cleanup step.

The aim of this project is to evaluate the performance and versatility of the Automate-Q40. A LC/QQQ was used to determine pesticide residues in fruit juices, particularly in apple juice. Pesticide residues were extracted from the apple juice by using the AutoMate-Q40. Quantification was based on matrix-matched calibration curves with the use of internal standard to ensure method accuracy. QC samples were evaluated at levels of 10, 50, 100 ng/g to ensure precision and accuracy of the AutoMate-Q40.

P84 Glyphosate Analysis in Soy Beans, Corn and Sunflower Seeds by HPLC with Post-Column Derivatization and Fluorescence Detection

Maria Ofitserova, Rebecca Smith, and Laszlo Torma
Pickering Laboratories Inc, Mountain View, CA, USA; maria_o@pickeringlabs.com

Glyphosate is a broad spectrum herbicide widely used around the world. Monitoring of Glyphosate in crops and water is mandated in many countries. We developed sensitive and robust HPLC method for analysis of Glyphosate in soy beans, corn and sunflower seeds. Simplified sample preparation procedure allows detecting Glyphosate on levels as low as 0.05 ug/g even in challenging matrices. Samples are extracted with water, partitioned with Methylene Chloride and cleaned using cation-exchange SPE columns. After HPLC separation Glyphosate is converted to fluorescence derivative by post-column derivatization. Recoveries for studied matrices ranged from 70% to 103%.

P85 Arsenic Speciation in Infant Rice Cereals using HPLC-ICP-MS

Rima Juskelis[1], Jack C. Cappozzo[1], and Jenny Nelson[2]
[1]**Institute for Food Safety & Health**, Illinois Institute of Technology, Bedford Park, IL, USA; jcappozz@iit.edu;
[2]Agilent Technologies, Santa Clara, CA, USA

Rice is one of the main sources of inorganic arsenic consumed in the current population's diet (1). Arsenic occurs naturally in the environment and can also be present as the result of human activity. Rice plants are especially efficient at soaking up arsenic from their environment because the flooded areas in which they are grown make it easier to take up arsenic compounds. Inorganic arsenic is a known carcinogen, and chronic exposure to low levels of arsenic has been linked to increased risk of bladder, lung and skin cancer, as well as Type 2 diabetes and cardiovascular disease (2). Rice samples from the United States have shown higher inorganic arsenic levels compared to other samples from around the world; infant rice cereal sold here is generally made from American rice (2). Brown rice tends to contain more arsenic than white rice. The United States and European Union have not set limits for arsenic in food products, including rice. China has a limit of 150 ng/g of inorganic As in rice (4). It is not clear how harmful arsenic in rice may be to human population and new data could aid in a risk assessment. Infants may be at higher risk because they are more susceptible to the harmful effects of arsenic than adults. The aim of this study was to conduct a survey of arsenic content in infant rice cereals (white, brown and mixed grain, both organic and non-organic) purchased at US supermarkets.

P87 Fast and Accurate Analysis of PBDEs in a Single Run, Including PBDE 209

Kory Kelly and Matthew Trass
Phenomenex, 411 Madrid Avenue; Torrance, CA 90501, USA; matthewt@phenomenex.com

Polybrominated Diphenyl Ethers (PBDEs) are aromatic and non-polar compounds that were used as flame retardants. After extensive usage, it was determined that these compounds are toxic and have been restricted or banned in many areas, including under the Stockholm Convention. PBDEs result in reproductive and other health effects, are toxic at low levels, and are subject to bioaccumulation. It is therefore important to measure these compounds at very low levels from environmental, food, and biological sources.

PBDEs consist of 209 individual conformations called congeners, which vary in toxicity. It's therefore important to measure and quantitate the individual congeners separately. To achieve the lowest levels of detection and highest degrees of confidence, high resolution gas chromatography with high resolution mass spectrometry (HRGC/HRMS) is used. Even using this advanced instrumentation, accurate separation of all congeners is difficult and requires long run times to provide enough resolution. In addition, not all congeners are stable and may degrade if activity exists in the system. One example is the most substituted congener 209, which often requires a separate analysis using a shorter column to reduce activity and provide sufficient results.
This work utilizes a new technology that allows for fast quantitation of toxic congeners with short run times, and includes the quantitation of congener 209 in the same analytical run. This eliminates the need for an extra instrument using an alternative column dimension to quantitate the necessary congeners. Comparison of existing methods and the proposed method are included highlighting improved sensitivity and shorter run times.

P88 A Fast and Effective Approach for Running EPA Method 539: Determination of Hormones in Drinking Water using SPE and LC/MS/MS

Ali Haghani[1], Andy Eaton[1], Jenny Wei[2] and Matthew Trass[2]
[1] Eurofins, 750 Royal Oaks Dr #100; Monrovia, CA 91016, USA
[2] **Phenomenex**, 411 Madrid Avenue; Torrance, CA 90501, USA; matthewt@phenomenex.com

Hormones have been found in a wide range of water supplies throughout the world. These compounds, including ethinyl-estradiol, the active ingredient in commonly prescribed birth-control medication, can cause detrimental effects to both aquatic life and humans. Because of the significant risk to human health and aquatic life, there is a rapidly growing public interest in monitoring these compounds. EPA method 539 was developed to address this large and growing public concern.

EPA Method 539 is a challenging analysis because not only does it require very low detection limits (0.1 part per trillion for some compounds), it also requires mass-spectrometer analysis in both positive and negative mode. In addition, more than 1,000 utilities are running EPA 539 in the United States alone. Therefore, it is extremely important to have a fast, accurate and reproducible analytical testing method.

This work follows the EPA method extraction protocol, in conjunction with an optimized LC/MS/MS method. The extraction protocol, after collection of the water sample, involves preservation and then solid phase extraction. The extract is then analyzed by LC/MS/MS under high pH mobile phase conditions. The optimized LC conditions result in a rapid analysis time of ~ 9 minutes. Aside from a fast analysis time, the analytical procedure results in excellent linearity and reproducibility (R^2 values of 0.99 for all compounds with 1/X quadratic fit). Because EPA 539 is such an important and widely used assay, the presented method is of great assistance to laboratories wanting to improve efficiency and productivity.

P89 Rapid Extraction and Determination of Select Anthelmintics in Milk by Liquid Chromatography/Mass Spectrometry (LC/MS)

Sarah E. McMullen,[1] Victor A. Vega,[1] Tony Williams,[1] and Frank J. Schenck[2]
[1]**US Food and Drug Administration,** Southeast Regional Laboratory, 60 Eighth St. NE; Atlanta, GA 30309, USA; sarah.mcmullen@fda.hhs.gov; [2]US Food and Drug Administration**,** Southeast Regional Laboratory, 60 Eighth St. NE; Atlanta, GA, 30309 USA; *retired*

A rapid screening method for the determination of four anthelmintics (Eprinomectin (EPR), Doramectin (DOR), Moxidectin (MOX), and Ivermectin (IVR)) in milk is presented. Milk samples are extracted in acetonitrile and the supernatant is diluted in basic water. Sample clean-up is performed via C_{18} column solid phase extraction (SPE). The SPE column is eluted with ethyl acetate, which is then collected and evaporated. The residue is reconstituted in 80% methanol and analyzed by liquid chromatography/mass spectrometry. For this study, exact mass LC/MS was utilized and validated. Instrument conditions for LC/tandem mass spectrometry were also developed are included for confirmatory purposes. Recoveries from fortified milk samples (1-10 ng/mL (ppb)) ranged from 96-118% (EPR), 83-112% (DOR), 57-76% (MOX), and 86-106% (IVR) (RSDs ≤ 20%). This simplified method presents several improvements over existing methods including simpler extraction, fewer consumables used and less hazardous waste produced.

P90 Analysis of Antibiotics in Food Matrix Using Liquid Chromatography High Resolution Accurate Mass Spectrometry

Jia Wang, Charles Yang, and Dipankar Ghosh
Thermo Fisher Scientific, San Jose, CA 95134, USA; jia.wang@thermofisher.com

The use of antibiotics in livestock farming has always been a concern because antibiotic residues may be present in food for human consumption. To comply with strict food safety standards, screening and quantitative methods for antibiotics are becoming

important. This poster describes a method for screening and quantitation of antibiotics in food samples using liquid chromatography coupled with a benchtop Orbitrap mass spectrometer, which provides high resolution accurate mass to unequivocally identify and quantify compounds without time consuming ms/ms optimization. An extraction method called "dilute-and-shoot" for this experiment was utilized. Water was added to the food samples and mixed using a vortex. Then organic solvent containing 1% formic acid was added, and then the sample was shaken for 1 h. The tube was centrifuged and the extract was transferred into an autosampler vial. 10 uL of the standards and extract was analyzed by liquid chromatography coupled with Orbitrap mass spectrometer. The mass spectrometer was set at different resolving power to evaluate the ability to resolve matrix from analytes. Calibration curves from 0.1 to 100 µg/kg were generated for the antibiotics analyzed. Depending on the antibiotics, the lowest concentration from the calibration curve can vary. The "dilute-and-shoot" sample preparation provides a fast extraction method. The resolving power of the Orbitrap instrument provides more confidence in resolving analytes from complex food matrix interferences, which resulted from the simplified extraction method. Furthermore, the method was developed using software with built-in workflows for streamlining method development and routine analysis.

P91 An Improved Screening Method for the Determination of Phthalate Residues in Various Commercial Milk Products by Bead Mixing and Supported Liquid Extraction Prior to LC-APCI-MS/MS

Frank Kero, Victor Vandell, Wendy Hartsock, Brad Nolt, and Elena Gairloch
Biotage, 10430 Harris Oak Blvd, Charlotte, NC 28269, USA; frank.kero@biotage.com

The migration of plastizers such as phthalic acid esters (phthalates) from the surface of food packaging materials into processed food materials and subsequent exposure to the consumer has long been an issue for public health. Continued interest in the biomonitoring of these compounds has inspired a number of method development strategies; however, classic methods are labor intensive and require multi-step time consuming efforts. For this reason, a novel sample extraction method has been developed. The extraction strategy for phthalates in commercial milk products was developed using a 3 stage sample preparation workflow prior to gradient LC-(+)APCI-MS/MS on a C4 column. A set of milk samples fortified with 5 phthalates was diluted with IPA and processed using an automated bead mixer to disrupt nonselective binding. The samples were then centrifuged and loaded onto a supported liquid extraction single use cartridge. This method was applied to commercial milk product variables to study the effect of fat content on relative recovery. To further evaluate method robustness, chocolate milk, butter milk and goat's milk were also evaluated. Typical relative recoveries ranged from 80-120% for diethyl-, dibutyl- , dipentyl-, octyl- and iso-nonyl-phthalate. The typical repeatability (%RSD) for n=7 replicates was <20%.

P92 Comparison of International Regulations for Pesticide Residues in Food

Eugene Chang, Irene Cassias, Olusegun Ajayi, and Shannon Lane
US FDA Pacific Regional Laboratory Southwest,; 19701 Fairchild Road, Irvine, CA 92612, USA;
Eugene.chang@fda.hhs.gov

There are more than 700 pesticides registered for use worldwide and around 2 million tons of pesticide were used each year. Based on the registration and use of pesticides still in production, countries and organizations have generated their own lists of pesticide to be tested. With globalization, monitoring pesticide residues in food from different parts of the world is a challenge. Over the last 50 years the legislation and enforcement groups have done a tremendous job using the resources they have to protect food safety for the general public. By combining the information gathered by these various groups we have collected a list of pesticide residues that can be used for monitoring, comparison, work planning and cost analysis. The objective of this work was to compare the regulations of Codex Alimentarius Commission, Europe Union, Japan and the United States and through this comparison combine the overlapped pesticides to generate a list. The list could be utilized in future method development and information management.

P93 The IR-4 Project: Celebrating 50 Years of Service to U.S. Farmers and Consumers

Johannes Corley, Daniel L. Kunkel, Jerry J. Baron, and Sherrilynn Novak
IR-4 Headquarters, Rutgers, The State University of New Jersey, 500 College Rd. East # 201 W., Princeton, NJ 08540, USA; jcorley@aesop.rutgers.edu

In 1963, the U.S. established the IR-4 project to facilitate the registration of pest management tools for specialty crops. Since then, IR-4 has been assisting U.S. producers of fruits, vegetables and other specialty crops manage their pest problems by providing safer and more effective chemical and biological mechanisms for insect, weed and disease control. Fifty years and more than 26,000 crop uses later, the IR-4 project still thrives and continues tackling even more challenging issues facing specialty crop growers with greater focus and vigor than ever. Today, several nations around the world are starting to emulate the IR-4 program in their own countries. The past, present and future of IR-4 project's efforts beginning 50 years ago and our plans for the future in keeping the U.S. food supply safe and plentiful and enabling you, the consumer to have plenty of delicious and nutritious fruits and vegetables on your table, will be on display.

P94 Persistence of three Herbicides in Soil Amended with Chicken Manure

George F. Antonious[1], Maifan Silitonga[1], Buddhi Gyawali[1], Louie Rivers[1], and Asmare Atalay [2]

[1]**Kentucky State University, College of Agriculture**, Food Science, and Sustainable Systems, Division of Environmental Studies, Frankfort, KY 40601, USA; george.antonious@kysu.edu;
[2] Virginia State University, Agricultural Research Station, Petersburg, VA 23806, USA

The retention and behavior of three herbicides, metribuzin, dacthal, and sethoxydim in soil and runoff water under three soil management practices were investigated. The main objectives were to: i) determine herbicides dissipation and half-life ($T_{1/2}$) values in soil amended with chicken manure (CM), soil amended with municipal sewage sludge (SS), and no-mulch (NM) unamended soil; ii) monitor the concentration of metribuzin, dacthal, and sethoxydim in runoff and infiltration water following natural rainfall; and iii) determine the impact of soil amendments on the transport of NO_3, NH_4, and P into surface and subsurface water. Half-life ($T_{1/2}$) values of metribuzin in soil were 44.3, 37.6, and 27.1 d in CM, SS and NM treatments, respectively. Similarly, $T_{1/2}$ values of dacthal and sethoxydim in soil were greater in CM and SS amended soil compared to native soil (NM). Addition of CM and SS to native soil increased water infiltration, lowering runoff water volume and herbicide residues in runoff following natural rainfall events.

P95 Quantitation of Antibiotics and Insecticides in Poultry Feed using Liquid Chromatography Tandem Mass Spectrometry

Tho Pham, [1]Ryan Picou,[2] Sarah Ruiz,[1] Cheryl Stephenson,[1] John Reuther,[1] Seyed Sadjadi,[3] and Lauryn Bailey[4]
[1]**Eurofins Analytical Laboratories**, 2315 N. Causeway Blvd; Metairie, LA 70001, USA; thopham@eurofinsus.com, johnreuther@eurofinsus.com; [2]Exxon, Baton Rouge, LA, USA; [3]Phenomenex, Torrance, CA, USA;[4]AB SCIEX, Framingham, MA, USA

Antibiotics have been added to poultry feeds in low doses to serve as growth promoters. These antibiotics accumulate in poultry feathers, which serve as a high protein additive ingredient that is added back into poultry feed. Continued use of antibiotics has created antibiotic-resistant micro-organisms, which has caused human health concerns. Another concern is residual insecticides, commonly and legally used during crop production, that remain in grains and related glutens, which are then used as components of animal feeds.

Quantifying low concentrations of antibiotics and insecticides in poultry feed is a major analytical challenge due to the inherent complexity of the sample matrix, which can include proteins, fats, carbohydrates, antimicrobials, emulsifiers, binders, pH control agents, pelleting agents and preservatives. An extraction, cleanup (SPE) and analysis (LC-MS/MS) method has been developed to meet this challenge. Fourteen antibiotics, which include fluoroquinolones, sulfonamides, amphenicols, macrolides, tetracyclines and quinolones, and four insecticides were analyzed in poultry feed. All compounds were analyzed in a single LC-MS/MS run using polarity switching. Method accuracy and reproducibility are demonstrated by evaluating fortified poultry feed samples over several days. Limits of Quantitation (LOQ) ranged from 3 to 12 ppb with the majority at 5 ppb. Recoveries of fortified feed sample were 70% overall, with the exception of the tetracyclines with recoveries of 50%.

P96 Study of Qualitative Screening Analysis of Organophosphorus Pesticides residues in foods using a GC-HRTOFMS with Fast GC Technique

Masaaki Ubukata, John Dane, and Robert B. Cody
JEOL USA, Inc.; 11 Dearborn road, Peabody, MA 01960, USA; mubukata@jeol.com

The fast gas chromatograph (GC) technique provides the capability to do high-throughput quantitative and qualitative analyses over a shorter time than traditional GC analyses. However, because the analytes elute from the column at a faster rate, this technique requires a high-speed data acquisition capability for the detector. Time-of-flight mass spectrometers (TOFMS) are a very good option for handling this requirement for the fast GC technique. Additionally, TOFMS systems can provide high mass accuracy and high mass axis stability so that accurate mass measurements can be easily performed even when using a single point mass calibration with an external reference ion. In this study, the fast GC analysis and accurate mass measurement of organophosphorus pesticides in pumpkin samples were investigated by using electron ionization GC-TOFMS. A frozen pumpkin sample was subjected to the QuEChERS cleanup method followed by a silica gel minicolumn extraction. A blank pumpkin extract solution was spiked with a standard mixture solution of 57 pesticides and then analyzed using the fast GC-TOFMS technique. All of the pesticides were easily detected in the extract samples with high mass accuracy (≤ 2mDa). Additionally, their recovery rates were shown to have standard deviation values (%) that were less than 10 %.Furthermore, the fast GC technique shortened the analysis time from approximately 24 minutes for traditional GC analysis to less than 12 minutes. These results clearly showed that the fast GC-TOFMS technique can become a powerful tool for the analysis of pesticide residues in foods.

P97 Practical Evaluation of a Multi-Residue Pesticide Screen in Fruits and Vegetables via GC-MS/MS

Steven Winter and Raymond Allum
Florida Department of Agriculture and Consumer Services, 3125 Conner Blvd Lab 3; Tallahassee, FL 32399, USA; Steven.Winter@FreshFromFlorida.com; Raymond.Allum@FreshFromFlorida.com

In this study we will provide a real-world working assessment of the overall efficiency and ruggedness of an optimized multi-residue pesticide screen. This method was used to screen USDA PDP (United States Department of Agriculture – Pesticide Data Program) samples. Findings are reported to PDP and then compiled into a national database. QuEChERS extracts of fruit and vegetable samples were analyzed via gas chromatography (GC) coupled with tandem mass spectrometry (MS/MS). The primary focus of this project was to produce a solution which could significantly improve the performance of the existing GC-MS/MS multi-residue pesticide screen

method without requiring any major changes to the extraction procedure. This project was initiated in direct response to the overall poor method performance of the GC-MS/MS screens previously utilized.

P98 Benefits of an Inert Flow Path for Pesticide Analysis in Food Matrices by GC/MS/MS

Limian Zhao and Mike Szelewski
Agilent Technologies Inc., 2850 Centerville Road, Wilmington, DE 19808, USA; limian_zhao8@agilent.com

Flow path inertness plays a critical role in the accuracy, precision, durability and consistency for the pesticides analysis in complicated sample matrices. The Agilent Inert Flow Path, including Ultra Inert column, inlet liner, and gold seal, plus UltiMetal Plus inert inlet, capillary flow technology (CFT) device and flexible metal ferrules, provides excellent surface inertness for the entire GC flow path and reduces negative impact to the target analytes caused by surface active sites. When using the Inert Flow Path, significant improvements were obtained, including higher response, better peak shape, better calibration curve linearity and entire flow path durability and consistency with multiple injections. In addition, the inert wool packed in the liner increased productivity by keeping non-volatile matrix interferences in the liner, thus extending column lifetime and reducing frequency of MS source maintenance.

In this study, trace level analysis of active and difficult pesticides in fruits and vegetables by GC/MS/MS was accomplished using Agilent Inert Flow Path. By using sandwiched injection, different matrix calibration curves were achieved by injecting one set of calibration standards with sandwich injection of matrix blank. This saved a lot of bench work to prepare the matrix spiked calibration standards, thus increased the consistency and decreased the potential errors caused by preparation mistakes. Six different matrices, including strawberry, orange, plum, onion, red pepper and spinach, were used for the system performance evaluation.
The results showed that the use of Inert Flow Path provided more accurate and precise quantitation results and longer durability for the pesticides analysis in food matrix. Superior sensitivity (1 ng/mL LOQ in matrix) and calibration curve linearity (over 1-100 ng/mL with $R^2 > 0.99$), excellent repeatability and performance stability (recoveries 80-110% and RSD < 15% over 70 injections) demonstrate that Agilent Inert Flow Path is an excellent choice for accurate pesticides analysis in fruits and vegetables by GC/MS/MS.

P99 Analysis of 19 PCB Congeners in Catfish Tissue using a Modified QuEChERS Method with GC-MS/MS

Narong Chamkasem and Tiffany Harmon
US FDA Southeast Regional Laboratory, 60 8th Street NE, Atlanta, GA, USA; narong.chamkasem@fda.hhs.gov

A simple and quick approach to determine 19 PCBs in catfish tissue is presented. A modified QuEChERS (quick, easy, cheap, effective, rugged, and safe) method employing high solvent to sample ratio (3 g sample/30 mL acetonitrile) was used to improve the extraction recovery of 19 PCBs. After salting out by shaking with 6 g anhydrous magnesium sulfate and 1.5 g sodium chloride and centrifugation to induce phase separation and partition pesticides to the acetonitrile phase, one mL of acetonitrile extract is pipetted into a 2-mL centrifuge tube containing 150 mg anhydrous magnesium sulfate, 150 mg primary secondary amine sorbent and 50 mg C-18 sorbent. The tube is mixed and centrifuged to absorb fat and fatty acid residue presence in the acetonitrile extract. The acetonitrile extract is injected into a gas chromatograph/tandem mass spectrometer (GC-MS/MS) using temperature programmable injector and column back flush program at the end of the run. The excellent sensitivity of GC-MS/MS allows injection of diluted sample to detect the pesticides in fatty sample without sample concentration. Column back flush program also keeps the GC column clean and minimizes instrument down time. The recovery and RSD for 19 analytes at 10, 100, and 300 ng/g (five replicates) are adequate at 92.2 ± 9.0, 87.3 ± 5.9, and 85.2 ± 6.9, respectively. The method is quick, uses minimum lab supplies, and provides excellent sensitivity with the LOQ less than 1 ng/g.

P100 Broad Scope Pesticide Screening in Food using GC Triple Quadrupole MS

David Steiniger, Juan Carmona, Paul Silcock, and Jason Cole
Thermo Fisher Scientific, 2215 Grand Ave Pkwy, Austin, TX 78728, USA; paul.silcock@thermofisher.com

The increased accessibility of high selectivity GC-MS has enabled more generic sample preparation in pesticide testing, allowing consolidation of multiple analyte lists and matrices into one method. GC-MS/MS is well suited to multi-residue analysis in a diverse range of matrices. However, as the number of targeted compounds increases, the complexity of method optimization increases and analytical performance becomes compromised. Presented is the use of smart instrument control and data processing software applied to GC-MS/MS analysis of >600 pesticides in matrix to mitigate analytical performance degradation through MS duty cycle optimization. Also discussed is combining this optimized targeted quantitation with general unknown analysis through fullscan/MRM.

Thermo Scientific TSQ 8000 Pesticide was used to construct a multi-analyte MRM method to monitor >600 pesticide residues using >1300 individual timed SRM (t-SRM) transitions. The low level quantitative performance was determined for a subset of ~50 pesticides spiked at 5 and 10 ppb in QuEChERS extracts of lettuce using the 600 pesticide method (referred to as “50/600 method”).

Data obtained using the 50/50 method show better detection limit performance than the 50/600 method even when using a t-SRM approach. However, early data has shown that the LOD performance of the 50/600 is within the EU MRLs specified and suggests this approach can be considered to monitor a large number of pesticide residues for official control while at the same time recording background prevalence of a wider range of residues in a “targeted screen”.

P101 Optimization of Simultaneous Derivatization for Rapidly Screening Banned Anabolic Steroids in Dietary Supplements by GC-MS-MS

Jason Tang and Timothy Baker
NSF International, Analytical Chemistry Laboratory, 789 Dixboro Road, Ann Arbor, MI, USA; tang@nsf.org
Studies have found that up to 25% of dietary supplements contain banned anabolic steroids or stimulants that are not specified on the products label. There is a great potential for athletes to unintentionally consume contaminated dietary supplements that can lead to a failed doping test. NSF International has established a special program called "Certified for Sports". The program helps ensure the product does not contain unacceptable quantities of contaminants for the recommended serving size through rigorous testing. Fast, easy and effective testing methods are needed to rapidly screen banned substances in dietary supplements. Gas chromatography-mass spectrometry (GC/MS) has been an essential technology for monitoring anabolic steroids in doping testing. The derivatization with trimethylsilylation of 84 anabolic steroids prohibited by World Anti-doping Agency was systematically optimized with silylation reagents N-methyl-N-trimethylsilyltrifluoroacetamide (MSTFA) and N-O-bis-(trimethylsilyl) trifluoroacetamide (BSTFA) in terms of selected catalysts [ammonium iodide (NH_4I) and trimethyliodosilane (TMIS)] and reducants { ethanethiol, dithioerythreitol (DTE), and 2-meraptoethanol}, solvent effects {acetonitrile, pyridine, ethyl acetate, dimethylformamide, tetrahydrofuran, and hexane}, and microwave power levels. This study showed that MSTFA was much more powerful than BSTFA. For steroids with keto groups or tertiary hydroxyl groups at carbon 17, catalysts must be added for completion of their derivatization with MSTFA due to steric hindrance in their structures. Two derivatization mixtures, 50% acetonitrile/pyridine (3:2, v/v) plus 50%MSTFA/TMIS/DTE (1000:5:2, v/v/w), or plus 50%MSTFA/TMIS/ethanethiol (1000:5:5, v/v/v), have been found to be the most effective reagents for simultaneous derivatization of all 84 steroids at 600W of microwave power for 1 min.

P102 A Simple Solution for Multi-Residue Analysis in Vegetables from Sample Prep to GC-MS/MS and LC-MS/MS Screening

Helen Sun, Zicheng Yang, and Kefei Wang
Bruker Daltonics, Inc., Chemical and Applied Markets, 3500 W Warren Ave, Fremont, CA 94538, USA; helen.sun@bruker.com

Tandem mass spectrometry coupled to chromatography, such as GC-MS/MS and LC-MS/MS, operated in MRM mode, has become the method of choice for targeted screening of multi-residue analysis in complex food matrix samples. A fast, easy and efficient sample preparation of food sample is the key to multi-residue pesticide MS analysis, which in fact still remains as a challenge. On the other hand, developing a multi-residue MRM method is labor-intensive and time-consuming. For production labs, it is always desired to have an easy-of-use software which integrates MRM method development flow to significantly speed up the method set-up.

In the current study, an improved QuEChERS protocol as an alternative to the conventional QuEChERS is employed for vegetable matrix extraction. It is easy, fast, and has comparable recovery rate. Three vegetables rice, spinach and avocado representing low moisture, high moisture, fatty sample respectively, were applied with the improved QuEChERS method for extraction. A pesticide mix containing 30 residues was spiked into each extracted matrix for calibration and analysis. The exacted matrix is diluted and directly shot into GC-MS and LC-MS for pesticide analysis, which largely simplifies sample prep and saves time. Good sensitivity on GC-MS/MS (1 ppb) and 0.1 ppb on LC-MS/MS were demonstrated. Great linearity was achieved as well. We also demonstrate MSWS 8.1 software with Compound Based Screening (CBS) workflow for fast MRM method development using Bruker Scion GC-MS/MS and EVOQ LC-MS/MS system. This study provides a complete solution for pesticide analysis in vegetable samples from sample prep to MS analysis.

P103 A Comparison of Hydrogen and Helium Carrier Gas for the Analysis of Pesticide Residues by GC/MS/MS

Ed George
Bruker Daltonics, Inc., 3500 West Warren Avenue; Fremont, CA 94538, USA; ed.george@bruker.com

In recent years, the gap between the supply and demand for helium gas has become increasingly tight. Shortages have forced facilities to ration use or even shut down. Helium has gone up in price by a factor of four from 2000 to 2012, and is expected to increase at a rate of 5 to 20% per year. Under these conditions, laboratories have considered hydrogen as an alternate carrier gas in GC/MS. The advantages of hydrogen are low cost, renewable resource (can be generated on demand), low viscosity and density (flattest Van Deemter curve for fast and efficient separations), and high diffusion coefficient.
However, converting GC/MS/MS pesticide residue methods to hydrogen carrier gas can present challenges in terms of laboratory safety, reactivity / absorption in both the injector and ion source, and decreased sensitivity. Some practitioners have tried connecting a hydrogen line to their GC/MS systems without making any changes to the method, only to find high background, poor chromatography, and severe mass spectral distortions, prompting them to abandon the endeavor.

The Bruker SCION TQ with Helium-Free package allows the user to operate safely with hydrogen carrier with minimal loss of sensitivity or spectral distortion. The unique axial ion source design with large turbo pumps are ideal for this low viscosity gas. In this study, data is compared between hydrogen and helium carrier for a group of 50 pesticides under the same column and flow conditions. Both hot splitless and pressure temperature vaporization (PTV) injection techniques are presented in terms of calibration, precision, and limit of detection.

P104 Ultra-sensitive Detection of Pharmaceutical and Personal Care Products (PPCP's) in Water by an Integrated On-Line Extraction-UHPLC-MS/MS System

Zicheng Yang, Helen Sun, and Kefei Wang
Bruker Daltonics, Inc., Chemical and Applied Markets, 3500 W Warren Ave, Fremont, CA 94538, USA; zicheng.yang@bruker.com

The occurrence of pharmaceutical and personal care products (PPCPs) in the environment water is of growing concern due to their potential harmful effects on environmental and human. The conventional methods based on the "template" of EPA 1694 method require pre-concentration of large volume of water sample and tedious SPE cleanup, followed by LC-tandem mass spectrometer analysis in order to achieve the low ng/L (ppt) level detection. In this study, we introduce an UHPLC system with an integrated online extraction (OLE) option coupled to a Bruker EVOQ Elite triple quadrupole mass spectrometer for detection of PPCPs in water. The Bruker Advance UHPLC OLE system consists of one binary pump and an integrated third pump for online sample preparation. The built-in online extraction requires no additional hardware assembly (LC pump and valve), and is able to detect 1-5 ppt of PPCPs in water by rapidly pre-concentrating 0.5 mL drinking water sample. The current work is focused on expanding the targeted PPCP list in one analytical run by the pre-concentration step and improving chromatographic separation. The final analysis method will be applied on the city-supplied drinking water samples and local lake water samples for PPCPs analysis.

P105 Analysis of Organophosphorus Pesticides in Baby Foods Using a Triple-Quadrupole GC/MS/MS

Laura Chambers, Richard Whitney, and Robert H. Clifford
Shimadzu Scientific Instruments, 7102 Riverwood Drive, Columbia, MD 21046, USA; lachambers@shimadzu.com

Contamination of food products with pesticides is a growing concern because of recognized adverse health effects, increased world-wide usage of pesticides, and increasing imports of raw foodstuffs from a large number of foreign sources. The concern is particularly acute for baby foods because of high vulnerability of infants to adverse health effects from numerous synthetic chemicals, particularly pesticides.

Gas chromatography mass spectrometry (GCMS) has been used extensively to quantify trace-level pesticides in food matrices; the most significant challenge has been matrix interference and achievement of meaningful health-based detection limits for the compounds of interest. The QuEChERS 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. But significant interferences still present a formidable problem for analysis of trace-level pesticides in foods, even after QuEChERS extraction and cleanup.

Triple quadrupole GC/MS/MS has emerged as the technique of choice for analysis of trace level contaminants in complex matrices. Operation of a triple quadrupole GC/MS/MS in the Multiple Reaction Monitoring (MRM) mode provides unmatched sensitivity and selectivity for detection and quantitation of targeted pesticides at low concentrations in the presence of interfering background.

This poster presents data illustrating the sensitivity of the Shimadzu GCMS-TQ8030, and its ability to detect trace level pesticide residues in baby food with essentially no matrix interference. Simultaneous acquisition of qualitative scan data and quantitative MRM data through the GCMS-TQ8030 unique scan/MRM mode is also discussed.

P106 A Quick Assay for the Quantitation of Deoxynivalenol in Grain Samples by Liquid Chromatography with UV Detection

Victor A. Vega, Michelle Young, and LaTisha Dorsey
US Food and Drug Administration, Southeast Regional Laboratory, 60 Eighth St. NE; Atlanta, GA, 30309, USA; victor.vega@fda.hhs.gov;

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.