

47th Florida Pesticide Residue Workshop

TradeWinds Island Grand Resort
St. Pete Beach, Florida
July 18-21, 2010







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2010 FLORIDA PESTICIDE RESIDUE WORKSHOP

July 18, 2010

Dear Attendees and Exhibitors,

Welcome to the 47th Annual Florida Pesticide Residue Workshop. We extend greetings to long-time attendees and first-time attendees, including international guests. And we thank our Exhibitors for your generous support of the Workshop.

We introduce several new things at FPRW this year, including a best scientific poster contest, as voted by attendees, and a whole-day technical session for Wednesday. Thank you for contributing more posters than ever for FPRW! The posters are displayed among the exhibits in the Pavilion all times that it is open (10:30 am Monday until noon on Wednesday), so be sure to view them all and make your vote with consideration.

Also, join us on **Sunday afternoon (2-5 PM)** for an informal discussion on technical and managerial issues, highlighted by a session on **Organic Analyses in Support of the Recent Gulf Coast Oil Spill**. This year, our MS Users Meeting on Tuesday afternoon features a debate between **Hans Mol** and **Jean-Phillipe Antignac** on different approaches to chemical residue identifications.

Monday morning, two internationally renowned scientists, **Michelangelo Anastassiades** and **Hans Mol**, give plenary lectures on **multiclass, multiresidue methods**. **Jonathan Friedan** follows with **How to Devise a "Greener" Lab and Facility**.

Monday afternoon starts with **Veterinary Drug Residues** chaired by Sherri Turnipseed and Perry Martos, and after a break, come back for **What You Don't Want to Know about QA and Lab Management but Can't Avoid**, moderated by Gail Parker and Kevin Armbrust. On Tuesday we go international with André de Kok and James Chang, for **Global Chemical Contaminant Conflicts… and Resolutions**. The afternoon session, with Kevin Armbrust and Gail Parker, answers an important question, **Out of the Analytical Bubble, Why Are We Analyzing Chemical Residues Anyway?** Wednesday morning, **Jana Hajslova** has a special talk on **Ambient Desorption Mass Spectrometry** for analyzing pesticides and other chemicals, and then the formal session **US Government in Action** starts, which is overseen by Lynda Podhorniak and Alaa Kamel. As noted earlier, we have a full Wednesday afternoon session, so please stick around for **Cream of the Crop Technical Talks** moderated by Alex Krynitsky and Steve Lehotay.

Vendor Seminars that feature the latest in scientific instrument and application developments are sprinkled throughout the FPRW program. Please make sure to visit the Exhibition for one-on-one discussions with vendors.

The **FPRW Organizing Committee Planning Meeting** is on Tuesday evening from 5:30 to 7:00 PM. FPRW is organized by scientific community volunteers, so come to the meeting, get involved, and help us insure a bright future for FPRW. FPRW is sponsored by FLAG Works, Inc., a not-for-profit organization.

The *Journal of Agricultural and Food Chemistry* is again offering to publish a special section affiliated with FPRW. If you have a manuscript to submit for possible publication, please inform Steve Lehotay, FPRW Program Chair. Submission deadline is September 1, 2010.

Door prizes will be consolidated this year to save time and provide a greater thrill in the audience. Arrive on time to oral sessions to get your ticket for drawings at the end.

The 2010 FPRW Organizing Committee thanks the Program Committee, especially its Chair, Steve Lehotay, for arranging interesting and internationally-appealing scientific sessions. Please enjoy, learn, discuss, and share.

Jack Cochran, Amy Brown, George Fong, Nicole Fisher, Kimberly Savage, Phil Wylie, and Jo Marie Cook



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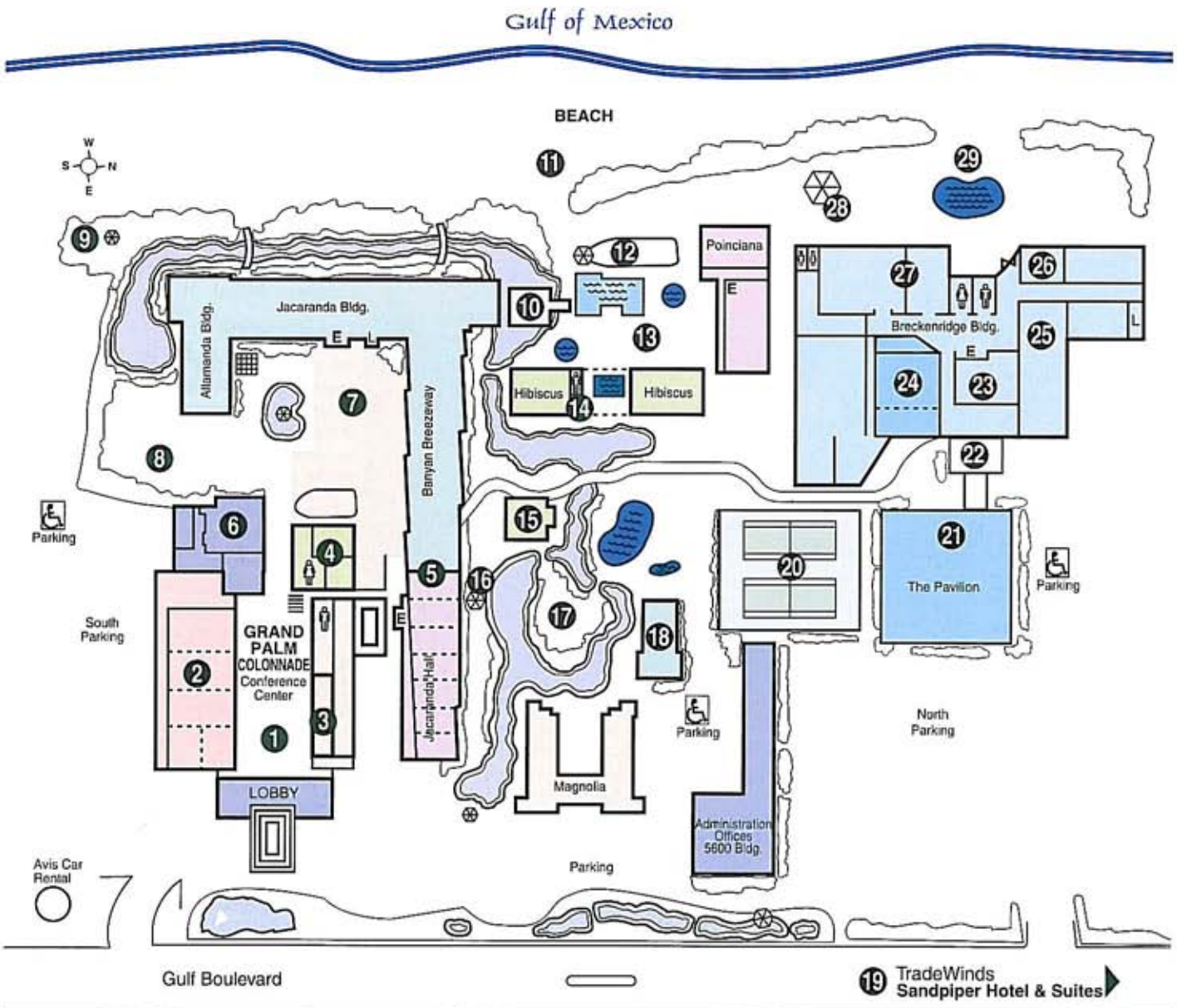
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Susan Eigen; Tifton, GA seigen@agr.state.ga.us
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Danny LeCompte; Auburn, AL lecomod@auburn.edu
Frank Schenck; Atlanta, GA fschenck@live.com
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Organizing Committee

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Web Site Content Manager: **Jo Marie Cook**; Florida Department of Agriculture and Consumer Services, Tallahassee, FL, USA cookj@doacs.state.fl.us
Vendor Outreach Chair: **Bill Holmes**; Mississippi State University, MS, USA wholmes@ra.msstate.edu

Program Committee (Session Organizers and Moderators)

Chair: **Steve Lehotay**; USDA Agricultural Research Service, Wyndmoor, PA, USA steven.lehotay@ars.usda.gov
Co-Chairs Elect/Moderators: **Lynda Podhorniak** and **Alaa Kamel;** EPA Analytical Laboratory, Ft. Meade, MD, USA podhorniak.lynda@epa.gov and kamel.alaa@epa.gov
Past-Chair/Moderator, **Alex Krynitsky**; FDA Center for Food Safety and Applied Nutrition, College Park, MD, USA alex.krynitsky@fda.hhs.gov
Poster Co-Coordinator: **Sarah McMullen**; FDA ORA Southeast Regional Laboratory, Atlanta, GA, USA sarah.mcmullen@fda.hhs.gov
Poster Co-Coordinators: **Paul Yang**; Ontario Ministry of the Environment, Etobicoke, ON, Canada paul.yang@ontario.ca.
Session Co-Organizer/Moderator: **Sherri Turnipseed**, FDA Animal Drugs Research Center, Denver, CO, USA sherri.turnipseed@fda.hhs.gov
Session Co-Organizer/Moderator: **Perry Martos**; University of Guelph, Guelph, ON, Canada pmartos@uoguelph.ca
Session Organizer/Moderator: **Gail Parker**; Florida Department of Agriculture and Consumer Services, Tallahassee, FL, USA parkerg@doacs.state.fl.us
Session Co-Organizer/Moderator: **André de Kok**; VWA - Food and Consumer Product Safety Authority, Amsterdam, The Netherlands andre.de.kok@vwa.nl
Session Co-Organizer/Moderator: **Jim Chang**; Thermo Fisher Scientific, San Jose, CA, USA james.chang@thermo.com
Session Organizer/Moderator: **Kevin Armbrust**; Mississippi State University, MS, USA armbrust@mscl.msstate.edu



Meeting Facilities

Grand Palm Colonnade 1
Island Ballroom 2
2nd Floor Meeting Rooms 3,4
Banyan Breezeway 5
Jacaranda Hall 5
Garden Courtyard 7
South Beach Lawn 9
Sea Breeze Deck 12
Pirate Island 17
Cypress Villa 18
The Pavilion 21
Horizons Portico 22
Horizons Meeting Room 24
Breck Deck 29

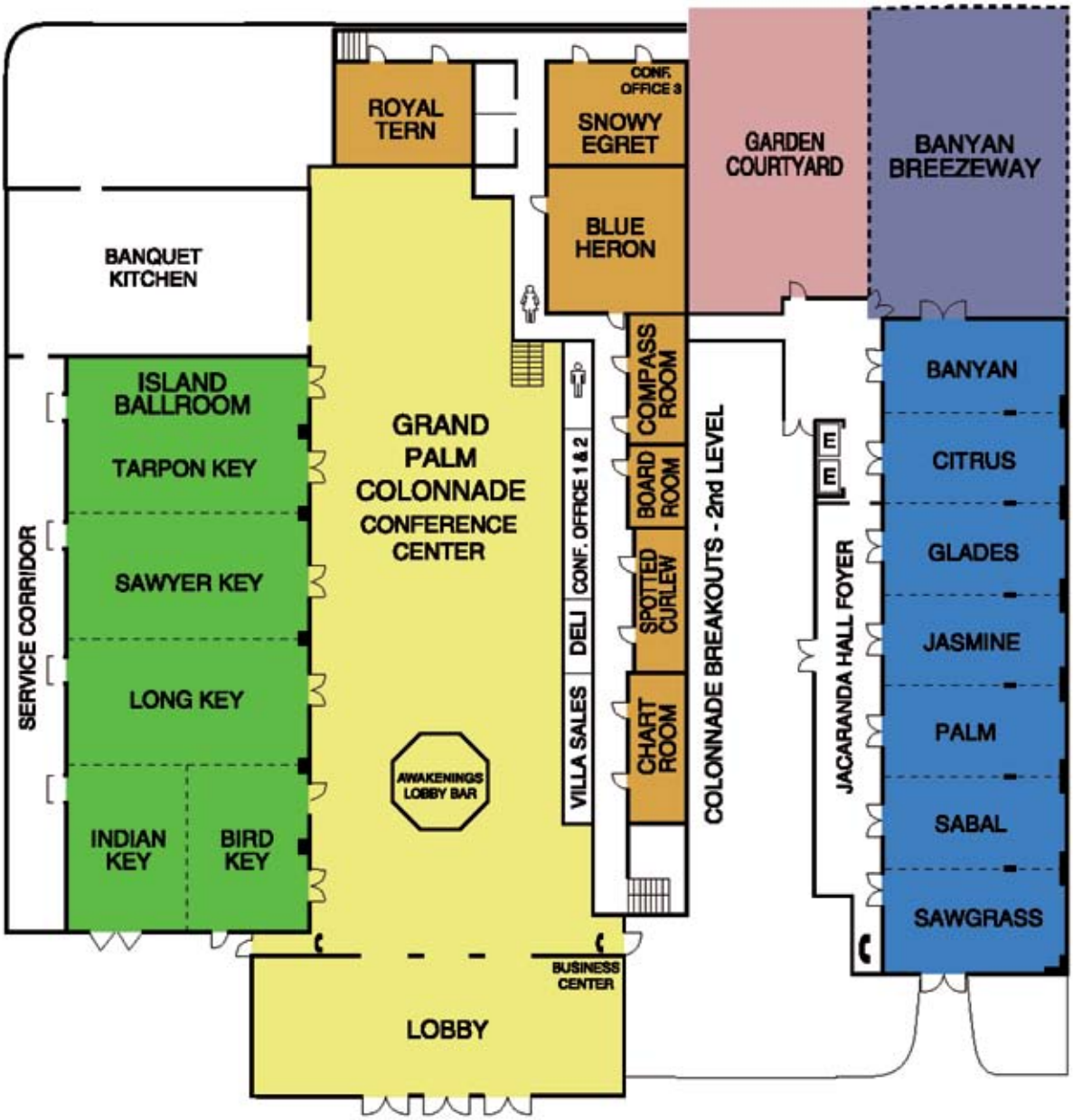
Dining & Entertainment

Awakenings Lobby Bar 1
Deli & General Store 3
Palm Court Bistro 6
Flying Bridge 10
RedBeard's Sharktooth Tavern . . . 15
RedBeard's Pirate Show 17
Coconuts Comedy Club 19
Pizza Hut Express 26
Old Meeting House Ice Cream . . 26
B.R. Cuda's Sports Bar 27
Bermudas Restaurant 27
Salty's Beach Bar 28
Sunset Celebration 28

Recreation & Shopping

Deli & General Store 3
Mini Golf 8
Pet Play Zone 8
Cabana Hut 11
High Tide Slide 11
Beachfront Terrace, Pool 13
Sauna, Covered Pool 14
Paddleboat Landings 16
KONK Club & Pool 18
Activities Center 18
Tennis Courts 20
Beaker's Tropical Outfitters 23
Body Works Spa & Fitness 25
Breck Adult Pool 29

Conference Center Map



Technical Session: Long Key / Bird Key and Indian Key Ballrooms

Exhibits, Posters, Reception: The Pavilion

Vendor Seminars: Banyan & Citrus Rooms (Jacaranda Hall)

Sunday Town Hall Discussion: Blue Heron

The Florida Pesticide Residue Workshop would like to thank the following companies for their support of the workshop!

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MEETING AT-A-GLANCE

Sunday, July 18, 2010

2:00 – 5:00 pm	**Sunday Town Hall Forum**	Blue Heron
5:00 pm →	Exhibition Set Up Begins	Pavilion
5:30 – 6:00 pm	Volunteers Orientation	Grand Palm Colonnade
6:00 – 8:00 pm	Registration	Grand Palm Colonnade
6:00 – 6:30 pm	Moderator Training	Long/Bird/Indian Key Ballrooms
6:00 pm →	Technical Session Setup	Long/Bird/Indian Key Ballrooms
6:00 pm →	Poster Board Set Up	Pavilion
7:00 – 8:00 pm	**Restek Evening Seminar**	Banyan & Citrus Rooms

Monday, July 19, 2010

All Day	Registration	Grand Palm Colonnade
10:30 am →	Exhibition & Posters	Pavilion
7:15 – 8:15 am	Early Morning Coffee	Pavilion
7:15 – 8:15 am	**Waters Breakfast Seminar**	Banyan & Citrus Rooms
8:30 – 10:30 am	Opening Oral Session	Long/Bird/Indian Key Ballrooms
10:30 – noon	Exhibition Opening	Pavilion
11:00 – noon	Poster Session A (authors)	Pavilion
12:15 – 1:15 pm	**Thermo Fisher Lunch Seminar**	Banyan & Citrus Rooms
1:30 – 3:00 pm	Veterinary Drug Analysis	Long/Bird/Indian Key Ballrooms
3:00 – 3:30 pm	BREAK (Exhibition & Posters)	Pavilion
3:30 – 5:00 pm	QA and Lab Management	Long/Bird/Indian Key Ballrooms
6:00 – 7:30 pm	Evening Reception	Pavilion

Tuesday, July 20, 2010

All Day	Registration	Grand Palm Colonnade
All Day	Exhibition & Posters	Pavilion
7:15 – 8:15 am	Early Morning Coffee	Pavilion
7:15 – 8:15 am	**Agilent Breakfast Seminar**	Banyan & Citrus Rooms
8:30 – 10:30 am	Global Chemical Contaminants	Long/Bird/Indian Key Ballrooms
10:30 – noon	Exhibition & Posters	Pavilion
11:00 – noon	Poster Session B (authors)	Pavilion
12:15 – 1:15 pm	**UCT, Inc. Lunch Seminar**	Banyan & Citrus Rooms
1:30 – 3:00 pm	Out of the Analytical Bubble	Long/Bird/Indian Key Ballrooms
3:00 – 3:30 pm	BREAK (Exhibition & Posters)	Pavilion
3:30 – 5:00 pm	Forum on Mass Spectrometry	Long/Bird/Indian Key Ballrooms
5:30 – 7:00 pm	Organizing Committee Meeting	Long/Bird/Indian Key Ballrooms
5:30 →	Beach Volleyball Game	On the Beach

Wednesday, July 21, 2010

All Day	Registration	Grand Palm Colonnade
Until noon	Exhibition & Posters	Pavilion
7:15 – 8:15 am	Early Morning Coffee	Pavilion
7:15 – 8:15 am	**Pickering Breakfast Seminar**	Banyan & Citrus Rooms
8:30 – 10:30 am	US Government in Action	Long/Bird/Indian Key Ballrooms
10:00 – 10:30 am	Exhibition & Posters	Pavilion
10:30 – noon	US Government in Action	Long/Bird/Indian Key Ballrooms
12:15 – 1:15 pm	**LECO Lunch Seminar**	Banyan & Citrus Rooms
1:30 – 2:50 pm	Technical Talks	Long/Bird/Indian Key Ballrooms
2:50 – 3:20 pm	BREAK	Grand Palm Colonnade
3:20 – 4:20 pm	Technical Talks	Long/Bird/Indian Key Ballrooms
4:20 – 4:30 pm	Closing (Poster Award)	Long/Bird/Indian Key Ballrooms


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GENERAL INFORMATION

Registration

Check in once at the registration desk at your earliest opportunity

Sunday - 6:00 – 8:00 pm
Monday - 7:30 am – 5:00 pm
Tuesday - 7:30 am – 5:00 pm
Wednesday – 7:30 am – 4:30 pm

KEY to Presentation Numbering System

Oral presentations are numbered **O-1, O-2, O-3, O-4,** (shut the door), **etc.**
Session A posters are numbered, **P-1, P-3, P-5, P-7,** (go to heaven), **etc.**
Session B posters are numbered, **P-2, P-4, P-6, P-8,** (who do we appreciate?), **etc.**

Poster Session (in The Pavilion)

Hang Posters Sunday evening after 6:00 pm or before 8:30 am on Monday
Take down posters by noon 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 Mon.
Poster Session B authors must be at their posters from 11:00 am – noon on Tue.

Poster Prize

Author(s) of Best Poster determined by popular vote of attendees will win the prize
Attendees must place their votes in the ballot box by noon on Wednesday

Exhibition

Monday - 10:30 am - 5:00 pm
Monday evening reception with cash bar 6:30 – 7:30 pm
Sponsored by TradeWinds Island Grand Resort

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. There will be morning and afternoon refreshment breaks each day. All Mon. and Tue. breaks, and the one on Wed. morning, will be served in the Exhibition Tent. On Wed. afternoon, the break will be served in the Grand Palm Colonnade.

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 W. George Fong. 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 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.

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 FPRW in 2010. Please inform Steve Lehotay (steven.lehotay@ars.usda.gov) in advance if you intend to submit an article. The manuscripts must be submitted electronically online on the journal's website by Sept. 1, 2010. Manuscripts will undergo the normal peer-review process to determine if it is acceptable for publication.

Copies of Presentations

Oral Presentations: Following the meeting, if time and resources permit, oral presentations will be posted on our website 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 PowerPoint 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.

Attendee List

After the meeting (approximately two weeks) an attendee list will be available, but only by request. Please contact W. George Fong at Gandwfong@comcast.net

Meeting Website

www.flworkshop.com - the website includes information on current and future FPRW meetings, as well as archives going back to 2005.

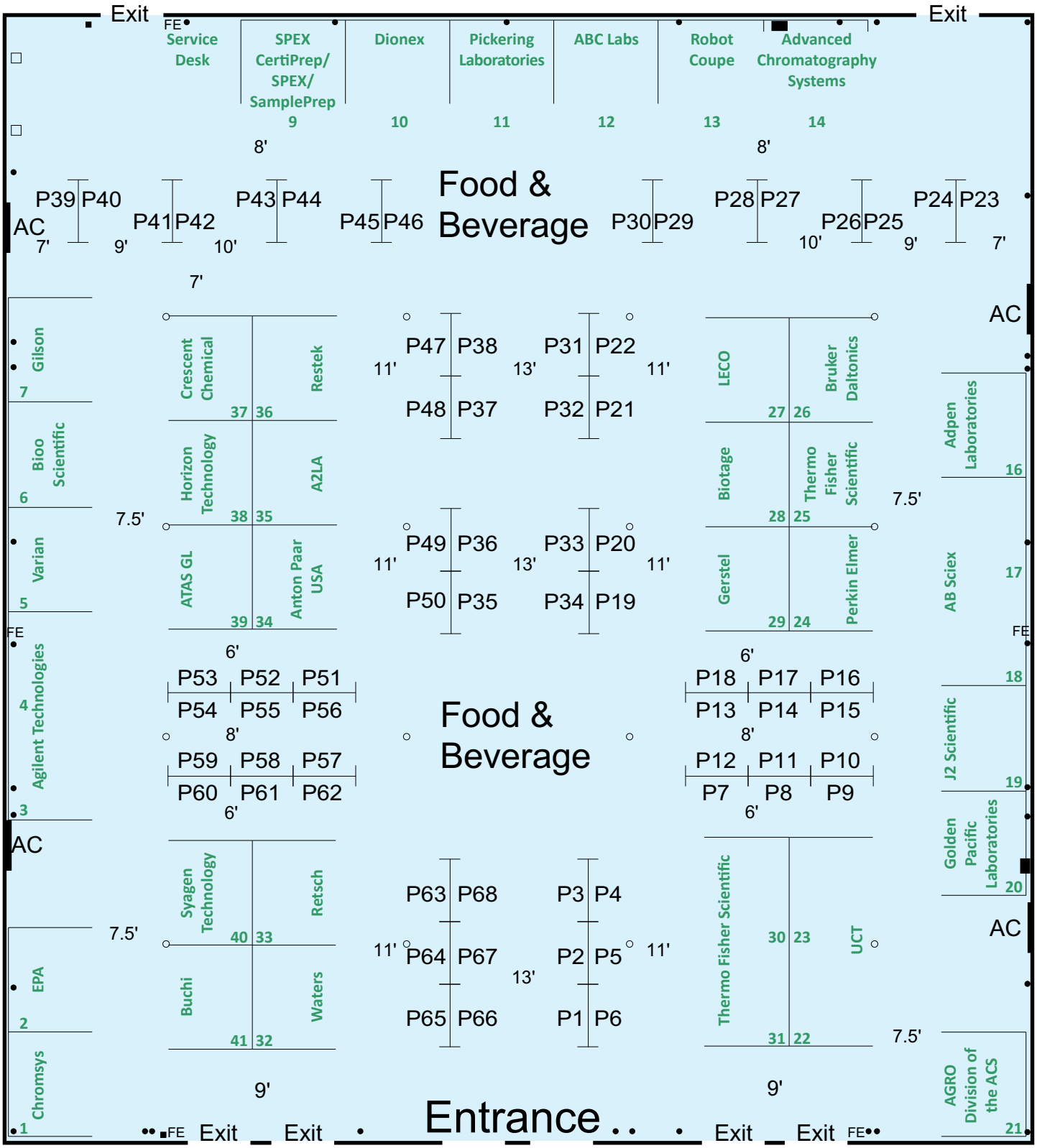
FUTURE MEETING DATES

2011 July 17-20
2012 July 16-19
2013 July 22-25
2014 July 21-24
2015 July 20-23

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

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Exhibition Map



Exhibitors

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VENDOR SEMINARS

Location: Banyan & Citrus Rooms (Jacaranda Hall)
Food and beverage provided by each company

Vendor Seminars: Please sign up at the meeting registration desk

V1. Sunday Evening, July 18, 2010, 7:00 - 8:00 pm: Restek

Enhancing That Certain Part of the GC Anatomy with QuEChERS and Cartridge SPE Cleanup for Pesticides in Dietary Supplements
Michelle Misselwitz, Jack Cochran, Jason Thomas, Julie Kowalski, and Rick Lake; Restek Corp., Innovations, 110 Benner Circle, Bellefonte, PA 16823; (814)353-1300, michelle.misselwitz@restek.com

The FDA recently announced that dietary supplement makers (e.g. vitamins, herbal and botanical pills, etc.) will have to test their products for purity. These supplements contain natural products where pesticides have been employed, so methods for pesticide analysis are needed, including sample extraction and cleanup, and instrumental techniques such as gas chromatography and mass spectrometry. The QuEChERS sample preparation approach offers a rapid way to prepare samples for analysis, but dietary supplement extracts can be so complex as to make trace-level pesticide determinations problematic. An elegant way to solve complex separation problems is to use comprehensive two-dimensional GC (GCxGC), especially when paired with a time-of-flight mass spectrometer (TOFMS). GCxGC applies two independent separations to a sample in one analysis by using a serial column configuration of different stationary phases separated by a thermal modulator. Separation results can be plotted as a retention plane. In this presentation, QuEChERS, cartridge SPE cleanup, and GCxGC-TOFMS results will be shown for pesticides in dietary supplements, including dandelion root, sage, "whole food nutrition" capsules, and "all natural" male enhancement products.

V2. Monday Breakfast, July 19, 2010, 7:15 - 8:15 am: Waters

An Overview of Advanced MS Technologies for Residue Screening; UltraPerformance Residue Analysis using UPLC TQ MS; Capability of QTof for Target and Non-Targeted Screening in Food, Water and Wildlife Samples
Timothy Jenkins; **Waters Corp. Chemical Analysis**, Atlas Park, Simonsway, Manchester, England, M22 5PP, **UK**; +44 161 435 4100, timothy_jenkins@waters.com

The use of mass spectrometry for pesticide residue analysis has changed a lot in the last 10-20 years, with multiresidue methods now being commonly used and approaches applied using other technologies to further increase the scope of analytical methods. This session will explore the most advanced tandem quadrupole and QTof technologies and how they can enhance the workflow for targeted and non-targeted approaches. Examples will be used for complex food matrices and direct injection of water samples using UPLC Xevo TQ MS, whilst with UPLC Xevo QTof approaches to enable broader scope screening, with the ability to identify unknown (non-targeted) peaks will be shown, using food, environmental and wildlife samples as worked examples.

V3. Monday Lunchtime, July 19, 2010, 12:15 - 1:15 pm: Thermo Fisher

EPA Method 1699: High Selective Multiresidue HRGC/HRMS Pesticide Analysis Applied to Food Samples
Heinz Mehlmann, Dirk Krumwiede, and Frank Theobald[2]; **Thermo Fisher Scientific**, Bremen, Hanna-Kunath Str. 11, Bremen, Bremen, 28199, **Germany**; 004942154930, heinz.mehlmann@thermofisher.com
2. Environmental Consulting Cologne, Cologne, **Germany**

Among recent official methods introduced by the U.S. Environmental Protection Agency (U.S. EPA) EPA Method 1699 can be found. This method is used for the determination of organochlorine, organophosphorus, triazine and pyrethroid pesticides in environmental samples by high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS) using isotope dilution and internal standard quantitation techniques. This EPA method is generally applied to aqueous, solid, tissue and biosolids matrices. The aim of our study was to extend the scope of applicable matrices for this method to include food samples. Furthermore the compatibility of this method with QuEChERS extracts has been investigated. The instrument sensitivity using EPA method was proven with standard measurements and resulted in the low pg/ul (ppb) range. For a number of components even sub ppb sensitivity can be achieved, which includes pesticides with strong tendency to fragment like Dieldrin, Aldrin, Endosulfans, and Endosulfan sulfate. The measurements of QuEChERS extracts from different food samples showed very good selectivity for most of the targeted pesticides with sensitivity well in the range requested by food regulations.

V4. Tuesday Breakfast, July 20, 2010, 7:15 - 8:15 am: Agilent

Analysis of Trace Contaminants in Foodstuffs using the Agilent 7000 Series Tandem Quadrupole GC/MS System

Chris Sandy; **Agilent Technologies UK**, Life Sciences and Chemical Analysis, 610 Wharfedale Rd, Winnersh Triangle, Wokingham, Berkshire, RG41 5TP, **UK**; 44-7836-236597, chris_sandy@agilent.com

The GC-MS detection, confirmation and quantitation of trace level agrochemicals in food-stuffs such as fruit, vegetables, herbs, spices and meat products can be impaired by co-extracted matrix components. Analysis by GC-MS/MS gives enhanced selectivity and sensitivity over selected ion monitoring (SIM) using a single quadrupole GC-MS system. The performance of a GC-MS/MS system for the quantitative analysis of pesticides and herbicides in spiked vegetable and meat extracts is shown. Data from food extracts analysed in MS-MS mode that demonstrates both quantitative accuracy and reproducibility for pesticides and herbicides spiked into samples at concentration levels ranging from 0.2 – 50 ppb. United States and European legislation specifies that polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzo-p-furans (PCDFs) in foodstuffs must be confirmed by high resolution capillary gas chromatography - high resolution mass spectrometry (HRGC-HRMS). In the event of a 'Dioxin incident' where contaminated products enter the Human food chain, it is very important to quickly establish both the source and extent of the contamination and, based on the analytical results, make a risk-assessment as to the potential hazard to Human health. This presentation gives a brief overview of a GC-MS/MS method and the data that can be obtained for the analysis of the 7 PCDD and 10 PCDF congeners specified in the legislation.

V5. Tuesday Lunchtime, July 20, 2010, 12:15 - 1:15 pm: UCT, Inc.

Chemistry of Solid Phase Extraction

Michael J. Telepchak, President and CEO; **UCT, Inc**, Marketing, 2731 Bartram Road, Bristol, PA 19007; (215)781-9255, mtelepchak@unitedchem.com

SPE, dispersive SPE, Solid Phase dispersion, filtration SPE or whatever else you would like to call it still depends on the same chemical principles. If you wish to get the best performance out of the technique, you need to understand the chemistry. This presentation will discuss the mechanisms encountered in SPE and how the chemistry affects the different mechanisms. Since C18 is the most popular phase in environmental analysis, we will discuss the chemistry of C18 columns and how the nature of the bonded phase is affected by the manufacturing process. The nature of more selective mechanisms, such as ion-exchange, will also be discussed and the affect of pH and distribution coefficients will be included.

V6. Wednesday Breakfast, July 21, 2010, 7:15 - 8:15 am: Pickering

Keys to an Analytical Method: Focus on Mycotoxins

Wendy Rasmussen and Bill Holmes[2]; **Pickering Laboratories, Inc.**, Technical Sales Dept., 1280 Space Park Way, Mountain View, CA 94043; (650)694-6700, wendyr@pickeringlabs.com
2. Mississippi State University, Starkville, MS

Efficient and reproducible sample cleanup are keys to an effective analytical method. GPC and Immunoaffinity clean-up are two techniques which are very effective for a wide range of matrices and analytes. We will continue the introduction of our LCTech Product line by describing our Mycotoxin analysis products, which include Immunoaffinity clean-up columns, high-performance ELISA kits for Aflatoxin and Ochratoxin A, and the versatile AcceCLEAN Automated column handling instrument. AcceCLEAN will handle three columns simultaneously, and can be used for many SPE applications in addition to Immunoaffinity clean-up. Furthermore, we offer individual or multi-residue mycotoxin methods utilizing Pickering Laboratories' Pinnacle PCX and UVE photochemical derivatization unit. These products can be used with a variety of matrices including corn, wheat, dried distillers grain, food and feeds. Bill Holmes, Director of Mass Spectrometry at Mississippi State University will also present their findings using the AflaCLEAN column and LC/MS/MS for the analysis of Aflatoxins in corn and dried distillers grain. He will present a statistical evaluation of the performance of their proposed method.

7. Wednesday Lunchtime, July 21, 2010, 12:15 - 1:15 pm: LECO

Comprehensive Two-Dimensional Gas Chromatography (GCxGC) Past and Present: *Exploring its Effective Use as a Routine Analytical Technique*

Joe Binkley and Mike Riley; **LECO Corp.**, Separation Science, 3000 Lakeview Ave., St. Joseph, MI 49085; (269)985-5730, joe_binkley@leco.com

This workshop will take a look at the early days of two-dimensional gas chromatography as well as current systems. The LECO comprehensive two-dimensional gas chromatography (GCxGC) systems operate with a dual column set. The two columns are connected in series via a dual-stage, quad-jet thermal modulator. The benefits of thermally modulated GCxGC include increased peak capacity, chromatographic resolution, and analyte detectability. After exploring some examples of these benefits, the workshop will discuss some of the many applications where GCxGC has moved its way into the mainstream as a more routine analytical technique. Applications covered will include the analysis of pesticides in foodstuffs, dietary supplements, and tobacco products.

MEETING PROGRAM

Sunday Afternoon July 18th

2:00 – 5:00 pm **Town Hall discussion forum on technical and managerial issues.**

2:00 – 3:00 pm Organic Analyses in Support of the Recent Gulf Coast Oil Spill -
Leaders of Discussion: **Dr. Ed Overton** and **Jack Cochran**
3:00 – 3:30 pm Anecdotes from Auditors and the Audited
3:30 – 4:00 pm Anecdotes from Company Phone Service Representatives
4:00 – 5:00 pm FDA's Pesticide Monitoring Program: Overview, Regulatory and Analytical Challenges, and Guidance for Private Labs Conducting Pesticide Residue Analysis

5:00 – 9:00 pm Technical Session Setup
5:00 – 10:00 pm Exhibition Set-up
5:30 – 6:00 pm Volunteers Orientation
6:00 – 6:30 pm Moderator Training
6:00 – 8:00 pm Registration
6:00 pm Poster Session Set up

7:00 – 8: 00 pm Vendor Seminar: **Restek Corporation**
V-1 Enhancing That Certain Part of the GC Anatomy with QuEChERS and Cartridge SPE Cleanup for Pesticides in Dietary Supplements - **Michelle Misselwitz**

Monday Morning July 19th

7:30 am - 5:00 pm Registration
Coffee will be served at 7:30-8:15 am

7:15 – 8:15 am Vendor Breakfast Seminar: **Waters Corporation**
V-2 1) An Overview of Advanced MS Technologies for Residue Screening;
2) UltraPerformance Residue Analysis using UPLC TQ MS;
3) Capability of QTof for Target and Non-Targeted Screening in Food, Water and Wildlife Samples - **Tim Jenkins**

8:30 – 10:30 am **Opening Oral Session** – Jack Cochran and Steve Lehotay, co-chairs

8:30 – 8:45 am Welcome and opening announcements

8:45 – 9:20 am Multiclass, Multiresidue Analysis of Pesticides Typically Analyzed in Single Analyte Methods -
O-1 **Michelangelo Anastassiades**; CVUA-Stuttgart, Fellbach, Germany

9:20 – 9:55 am Multiclass, Multiresidue Analysis of Pesticides, Veterinary Drugs, Environmental Contaminants, and Mycotoxins -
O-2 **Hans Mol**; RIKILT, Wageningen, The Netherlands

9:55 – 10:30 am How to Devise a "Greener" Lab and Facility –
O-3 **Jonathan Friedan**; Ballinger, Philadelphia, PA

10:30 am – noon Exhibition Opening (with ribbon cutting ceremony); Exhibition will be open rest of day
Danishes, coffee, and muffins will be served at 10:30 am

11:00 am – noon **Poster Session for rest of day – Group A authors at posters**
Sarah McMullen and Paul Yang co-coordinators of poster sessions

Monday Afternoon July 19th

12:15 – 1:15 pm Vendor Lunch Seminar: **Thermo Fisher Scientific**
V-3 EPA Method 1699: High Selective Multiresidue HRGC/HRMS Pesticide Analysis applied to food samples -
Heinz Mehlmann

1:30 – 3:00 pm Technical Session – **Veterinary Drug Residue Analysis**
Sherri Turnipseed and Perry Martos (co-chairs)

1:30 – 2:00 pm **O-4** Controlling Hormonal Residues in Animal Biological Matrices: from Conventional Targeted Methods to Emerging Global "Omic" Approaches –
Jean-Philippe Antignac; National Veterinary University of Nantes, France

2:00 – 2:30 pm **O-5** Analysis of Antiviral Drug Residues in Poultry and Swine by LC-MS/MS –
Mary Carson; FDA Center for Veterinary Medicine, Laurel, MD

2:30 – 3:00 pm **O-6** Improving Analytical Efficiencies in Single-Analyte Methods for Veterinary Drug Residues in Foods of Animal Origin –
Bryn Shurmer; Canadian Food Inspection Agency, Saskatoon, Canada

3:00 – 3:30 pm Break for Exhibition and Posters

3:30 – 5:00 pm Oral Session – **What You Don't Want to Know About QA and Lab Management but Can't Avoid** -
Gail Parker, chair (Kevin Armbrust, co-chair)

3:30 – 4:00 pm **O-7** What You Always Wanted to Know About Root Cause Analysis and Managements Review, but Were Afraid to Ask -
Arlene Fox; AOAC International, Gaithersburg, MD

4:00 – 4:30 pm **O-8** ISO 17025, Section 5.9, or How I Learned to Stop Worrying and Love the Bomb -
Gail Parker; FL Dept. of Agriculture and Consumer Services, Tallahassee, FL; USA

4:30 – 5:00 pm **O-9** Statistics Without Math -
Barry Nussbaum; EPA Office of Environmental Information, Washington, DC

6:00 to 7:30 pm Evening Reception sponsored by TradeWinds

Tuesday Morning July 20th

All Day Registration, Exhibition, and Poster Session

7:30 – 8:15 am Early Morning Coffee in Exhibition Hall

7:15 – 8:15 am **V-4** Vendor Breakfast Seminar: **Agilent Technologies**
Analysis of Contaminants in Foodstuffs using the 7000 Series Tandem Quadrupole GC-MS System - **Chris Sandy**
Recent Developments in LC-MS/MS Analysis of Food Contaminants - **Peter Stone**

8:30 – 10:30 am Oral Session – **Global Chemical Contaminant Conflicts … and Resolutions**
André de Kok and James Chang, co-chairs

8:30 – 9:00 am **O-10** Recent Developments in European Pesticide Registration and Impact on Global Trade -
Monika Bross; BASF, Limburgerhof, Germany

9:00 – 9:30 am **O-11** Challenges for Governmental Pesticide Laboratories in Brazil/Latin America -
Ionara Pizzutti; Federal University of Santa Maria, Brazil

9:30 – 10:00 am **O-12** Pre-Harvest Monitoring and Management of Pesticide Residues in India -
Kaushik Banerjee; National Research Centre for Grapes, Pune, India

10:00 – 10:30 am **O-13** Monitoring of Pesticide Residues in Export Products of Plant Origin in China –
Jianhua Wang – Shandong Exit & Entry Inspection and Quarantine Bureau, Qingdao, China

10:30 am – noon Exhibition and Posters (all day); Danishes, coffee, and muffins will be served at 10:30 am

11:00 am – noon **Group B authors at posters**

Tuesday Afternoon July 20th

12:15 – 1:15 pm Vendor Lunch Seminar: **UCT, Inc.**
V-5 The Chemical Principles of Solid Phase Extraction in Residue Analysis –
Michael Telepchak

1:30 – 3:00 pm Oral Session – **Out of the Analytical Bubble, Why Are We Analyzing Chemical Residues Anyway?**
Kevin Armbrust, chair (Gail Parker co-chair)

1:30 – 2:00 pm Pesticide Residue Analysis: Fifty Years of Accomplishments and Continuing Challenges
O-14 **James Seiber**; University of California at Davis, CA

2:00 – 2:30 pm Measuring Environmental Exposures: the Other Key to Risk Assessment -
O-15 **Keith Solomon**; University of Guelph, Canada

2:30 – 3:00 pm Helping the Farmer Helps the Regulator, Retailer, Consumer, and Environment: Everybody Wins -
O-16 **Danit Klein**; Lab-Path, Ziona, Israel

3:00 – 3:30 pm Break for Exhibition and Posters

3:30 – 5:00 pm Forum on Mass Spectrometry: **Hans Mol** and **Jean-Philippe Antignac** will discuss in moderated debate format different approaches to make chemical residue identifications using MS techniques;
Moderator: Sherri Turnipseed

5:30 – 7:00 pm Organizing Committee Meeting

5:30 pm – ??? Beach Volleyball Game

Wednesday Morning July 21st

Until noon Exhibition

Until noon Poster Session

7:30 am – 4:30 pm Registration

7:15 – 8:15 am Vendor Breakfast Seminar: **Pickering Labs**
V-6 Keys to an Analytical Method: Focus on Mycotoxins
Wendy Rasmussen and **Bill Holmes** (Mississippi State University)

7:30 – 8:15 am Early Morning Coffee in Exhibition Hall

International Travel Accommodation Talk:
8:30 – 8:50 am Ambient Desorption Mass Spectrometry: A New Challenge in Rapid Pesticide Residues/
O-17 Other Chemicals Control?
Jana Hajslova, Institute of Chemical Technology; Prague, Czech Republic

8:50 am – noon Oral Session - **US Government in Action**
Lynda Podhorniak and Alaa Kamel, co-chairs

8:50 – 9:20 am What's New with the Pesticide Residue Program in the FDA?-
O-18 **Christopher Sack**; FDA Total Diet Study Research Center, Lenexa, KS

9:20 - 9:40 am Multiresidue Analysis of Pesticides in Botanical Dietary Supplements, Herbals and Dried
O-19 Botanicals by GC-MS and LC-MS Techniques -
Jon Wong; FDA Center for Food Safety and Applied Nutrition, College Park, MD

9:40 - 10:00 am Determination of 200 Pesticides in 24 Food Matrices by Liquid Chromatography-Tandem Mass Spectrometry -
O-20 **Kai Zhang**; FDA Center for Food Safety and Applied Nutrition, College Park, MD

10:00 – 10:30 am Exhibition and Posters - Danishes, coffee, and muffins will be served at 10:00 am
Final Votes by the Attendees for Best Poster are Due by Noon in the Ballot Box

10:30 – 11:00 am Changes in the FSIS Field and Lab Operations on Residue Analysis –
O-21 **Terry Dutko** - USDA Food Safety Inspection Service, St. Louis, MO

11:00 – 11:30 am 20-Year Retrospective and Update of the Pesticide Data Program -
O-22 **Diana Haynes**; USDA Agricultural Marketing Service, Manassas, VA

11:30 am – noon Risk Assessment and Monitoring Data - Historical Aspects -
O-23 **Susan Hummel**; EPA Health Effects Division, Washington, DC

Wednesday Afternoon July 21st

afternoon Posters take down

afternoon Exhibition take down

12:15 – 1:15 pm Vendor lunch seminar: **LECO Corporation**
V-7 Comprehensive Two-Dimensional Gas Chromatography (GCxGC) Past and Present: Exploring its Effective Use as a Routine Analytical Technique -
Joe Binkley and **Mike Riley**

1:30 – 4:30 pm Oral Session - **Cream of the Crop Technical Talks**
Alex Krynitsky and Steve Lehotay, co-chairs

1:30 – 1:50 pm Targeted GC Pesticide Analysis at FDA: Transitioning to GC/MS/MS from GC/MS with
O-24 Selected Ion Monitoring –
Greg E. Mercer; FDA Pacific Regional Laboratory Northwest, Bothell, WA

1:50 – 2:10 pm Practical Tools for GC-MS Analysis of Pesticides in Complex Matrices
O-25 **Katerina Mastovska**; Covance Laboratories, Greenfield, IN

2:10 – 2:30 pm Development of a Sample Preparation Method and Analytical Assay using LC-MS/MS
O-26 for Quantitation of 10 Mycotoxins in a Single Analysis
Lauryn E. Bailey – Eurofins Scientific, Metairie, LA

2:30 – 2:50 pm Flow Injection Mass Spectrometry for High-Throughput Pesticide Residue Measurements
O-27 **Sergio C. Nanita** - DuPont Crop Protection, Newark, DE

2:50 – 3:20 pm Break

3:20 - 3:40 pm Organochlorine Pesticides Determination in Strawberries and Jam using
O-28 QuEChERS Extraction and GC-MS/MS –
Valentina Domingues – Instituto Superior de Engenharia do Porto, Portugal

3:40 – 4:00 pm Pesticide Residue Analysis of Catfish for the USDA-Pesticide Data Program (PDP)
O-29 **Jonathan Barber** – USDA Agricultural Marketing Service, Gastonia, NC

4:00 – 4:20 pm Determination of Three Neonicotinoids and their Metabolites in Pollen and Nectar of
O-30 Field Treated Pumpkin to Assess their Potential Exposure to Honey Bees
Alaa Kamel – EPA Office of Pesticide Programs, Fort George G. Meade, MD

4:20 – 4:30 pm **Poster award at close of meeting, thanks, and farewells**

Meeting Adjourns

Next Year Dates: July 17 - 20, 2011, TradeWinds Island Grand Resort, St. Pete Beach

POSTERS

Authors of odd numbered posters stand by their posters on Monday from 11 am to noon
Authors of even numbered posters stand by their posters on Tuesday from 11 am to noon

P-1 **Dissipation of Dimethazone Residues in Agricultural Field**
George F. Antonious; **Kentucky State University**, Frankfort, KY

P-2 **LC-ESI-MSMS Analysis of Toxic Industrial Chemicals from UV/TiO2 Photocatalytic Oxidation**
William C. Heiser, et al.; **Advanced Concepts and Technologies International**, Waco, TX

P-3 **Translocation of ^{14}C-Glyphosate Between Seedlings of Brachiaria brizantha and Coffee (Coffea arabica) and Citrus (Citrus limonia)**
Paulo Alexandre de Toledo Alves, et al.; **Centro de Energia Nuclear na Agricultura**, Brazil

P-4 **LC-MS/MS Analysis of Triazine Pesticides in Drinking Water using a New Software for Streamlined Method Development**
Charles Yang, et al.; **Thermo Fisher Scientific**, San Jose, CA

P-5 **Large Injection Volumes and Online Pre-Concentration for the Analysis of Pesticides in Beverage Utilizing High Resolution Accurate Mass LCMS**
Kristi Akervik, et al.; **Thermo Fisher Scientific**, San Jose, CA

P-6 **Qualitative Aspects in the Analysis of Pesticide Residues in Fruits and Vegetables using Fast, Low-Pressure GC/TOF**
Urairat Koesukwiwat; **USDA Agricultural Research Service**, Wyndmoor, PA

P-7a **Performance Evaluation of LC-Single Stage Orbitrap-MS for Screening of Residues and Contaminants in Food and Feed**
Hans Mol; **RIKILT, Institute of Food Safety**, The Netherlands

P-7b **Target Screening and Quantification Software for GCxGC-TOF-MS Data**
Hans Mol; **RIKILT, Institute of Food Safety**, The Netherlands

P-8a **Development of Immunochemical Methods (ELISA and Immunosensor) for Determination of Paraquat Residues in Food Samples**
Hans Mol; **RIKILT, Institute of Food Safety**, The Netherlands

P-8b **Direct Analysis of Dithiocarbamates by DESI-MS/MS**
Hans Mol; **RIKILT, Institute of Food Safety**, The Netherlands

P-9a **Direct Analysis of Dithiocarbamates by Ambient Mass Spectrometry Employing a Direct Analysis in Real Time (DART) Ion Source**
Tomas Cajka, et al.; **Institute of Chemical Technology**, Czech Republic

P-9b **Comprehensive Two-Dimensional Gas Chromatography–Time-of-Flight Mass Spectrometry (GC×GC–TOFMS) for the Determination of Multiple Pesticide Residues in Baby Food**
Tomas Cajka, et al.; **Institute of Chemical Technology**, Czech Republic

P-10 **Automated Purification Method for OCPs in Sediment using a SPE-GPC-VAP System**
T.K. Dobbs; **J2Scientific**, Columbia, MO

P-11 **MSPD Determination of Pesticide Residues in Beeswax**
Silvina Niell, Veronica Cesio; **Pharmacognosy & Natural Products**, Uruguay

P-12 **Straightforward Determination of 40 Organophosphorous Pesticides in Raw Wool using Microwave Assisted Extraction and GC/FPD Analysis**
María V. Cesio, Silvina Niell, et al.; **Pharmacognosy & Natural Products**, Uruguay

P-13 **Multi-Mycotoxin Analyses, including Cyclopiazonic Acid, in Various Grains by Mini-Column LC-MS/MS**
Perry A. Martos, et al.; **University of Guelph**, Ontario, Canada

P-14 **Determination of Pesticide Residues in Vegetables and Fruit Juices using Triple Quadrupole LC-MS/MS**
Yang Chen, et al.; **National Center for Food Safety Technology/IIT**, Summit-Argo, IL

P-15 **Flexible, Automated Sample Processing System for Analysis of Drinking Water Using SPE Cartridges and Disks**
Thomas K. Dobbs, et al.; **J2Scientific**, Columbia, MO

P-16 **Ultra-Fast LC/MS/MS Analysis of Acidic Herbicides in Water Samples by Direct Injection: Evaluation of Different MRM Techniques Combined with Polarity Switching**
Rolf Kern; **AB SCIEX**, Foster City, CA

P-17 **Simultaneous Screening Method for Residual Veterinary Drugs in Milk and Honey Using LC-MS/MS after QuEChERs Clean-Up**
YunYun Zou, et al.; **AB SCIEX**, Ontario, Canada

P-18 **Pesticide Screening and Quantitation in Cotton Goods**
Brian Shofran, et al.; **AB SCIEX**, Fort Myers, FL

P-19 **Determination of Aminopyralid in Soil and Vegetation by Solid-Phase Extraction and LC-ESI/MS/MS**
Jona Verreth, et al.; **Montana Department of Agriculture**, Bozeman, MT

P-20 **Automated QuEChERS Extraction for the Confirmation of Pesticide Residues in Foods using LC/MS/MS**
Virgil Settle, et al.; **GERSTEL, Inc.**, Linthicum, MD

P-21 **Optimized Extraction of Paraquat and Diquat from Various Matrices Prior to LC-MS/MS Analysis**
Russ Katz; **Biotage**, Charlotte, NC

P-22 **Quantitation of Pesticides in Flue-Cured Tobacco by Comprehensive Two-Dimensional Gas Chromatography-Time of Flight Mass Spectrometry (GCxGC-TOFMS)**
Joe E. Binkley; **LECO Corporation**, St. Joseph, MI

P-23 **NOT PRE-DESIGNATED**

P-24 **Transfer and Speed-up of Methods to Fused-Core™ Particle Columns - EPA Method 1694**
Ed Mauney; **Supelco/Sigma-Aldrich**, Statesboro, GA

P-25 **Method for Analysis of Organophosphorous Pesticides in Produce using Acetonitrile Extraction, Dual-Layer Carbon-Aminopropylsilica Cleanup and GC-NPD Detection**
Ed Mauney; **Supelco/Sigma-Aldrich**, Statesboro, GA

P-26 **Get Connected to The Water Laboratory Alliance!**
Marsha Pryor; **Pinellas County Utilities Laboratory**, Largo, FL

P-27 **Determination of Organophosphate and Triazine Pesticides in Water by GC-ECD**
Valentina Fernandes Domingues; **ISEP-REQUIMTE**, Portugal

P-28 **Determination of Organochlorine Pesticides from Organic and Integrated Pest Management Farming Strawberries using QuEChERS**
Vírginia Cruz Fernandes, et al.; **Centro de Investigação em Química/REQUIMTE**, Portugal

P-29 **Determination of Endocrine Disruptors in Portuguese Rivers**
Virgínia Cruz Fernandes, Valentina Fernandes Domingues; **ISEP-REQUIMTE**, Portugal

P-30 **GPC Clean-Up for Pesticide Residues in Fatty Food**
Wendy Rasmussen, et al.; **Pickering Laboratories**, Mountain View, CA

P-31 **Student Internships at the US FDA: A Unique Opportunity**
Yelena Karaseva, et al.; **FDA ORA Northeast Regional Laboratory**, Jamaica, NY

P-32 **Inter-laboratory Validation of a Modified QuEChERS and LC-MS/MS Method for Analysis of >250 Pesticide Residues in Six Fatty Food Products**
Yi Lin, et al.; **FDA ORA Pacific Regional Laboratory Southwest**, Irvine, CA

P-33 **Rapid Dispersive SPE Multiresidue Method for Analysis of Polar and Nonpolar Pesticides using Acetone**
Olusegun J Ajayi, et al.; **FDA ORA Pacific Regional Laboratory Southwest**, Irvine, CA

P-34 **Monitoring for Nontargeted Veterinary Residues in Food Samples using Quadrupole Time-of-Flight (Q-TOF) LC/MS**
Sherri B. Turnipseed, et al.; **FDA Animal Drugs Research Center**, Denver, CO

P-35 **Tubes or Tips?**
Lynda Podhorniak; **EPA Analytical Chemistry Branch**, Fort Meade, MD

P-36 **Disposable Pipette Extraction (DPX) Tips for Rapid Cleanup for Simultaneous Analysis of Melamine and Cyanuric Acid using GC/MS/MS and LC/MS/MS**
William E. Brewer, et al.; **University of South Carolina**, Columbia, SC

P-37 **Determination of Nitrofuran Metabolites in Shrimp by LC/MS/MS**
Lin Ye, et al.; **FDA ORA Southeast Regional Laboratory**, Atlanta, GA

P-38 **Development of a Multi-Residue Method for the Determination of Steroid Hormones in Bovine Muscle using LC-MS/MS**
Tricia L Johnson; **Food & Drug Administration**, Office of Research, Laurel, MD

P-39 **NOT PRE-DESIGNATED**

P-40 **Evaluation of Several Columns and Solvents for Post-Extraction GPC Clean-Up of Fish Tissue Prior to PBDE or PCB Analysis**
Michael Halvorson, et al.; **Gilson, Inc.**, Middleton, WI

P-41 **The MaxSignal® Trifluralin ELISA Kit is a Competitive Enzyme Immunoassay for the Quantitative Analysis of Trifluralin in Fish and Meat**
Lance Ford, et al.; **Bioo Scientific Corp.**, Austin, TX

P-42 **Determination of Selected Persistent Organic Polluting Pesticides in Water using Automated SPE and GC/ECD**
Robert S. Johnson, and Bob Riggs; **Horizon Technology**, Salem, NH

P-43 **Optimization of Solid-Phase Extraction Combined with Centrifugation for the Determination of Polycyclic Aromatic Hydrocarbons in Water**
Abir Kouzayha, et al.; **Lebanese Atomic Energy Commission**, Lebanon

P-44 **Effect of Seed Weight on Pesticide Residues Estimation in Stone Fruits**
Kazuaki Iijima; **The Institute of Environmental Toxicology**, Japan

P-45 **Large Volume Injection for Gas Chromatography using a Commercially-Available Unmodified Splitless Injector**
Jack Cochran; **Restek Corporation**, Bellefonte, PA

P-46 **Large Volume Injection on Split/Splitless Inlets "The Pauper's Upgrade"**
Lee Miller, et al.; **Florida Dept. of Agriculture and Consumer Services**, Tallahassee, FL

P-47 **QuEChERS Approach with GCxGC-ECD and GC-TOFMS for the Analysis of Pesticides in Dried Ginseng**
Michelle Misselwitz, et al.; **Restek Corporation**, Bellefonte, PA

P-48 **Analysis of Polycyclic Aromatic Hydrocarbons in Infant Formula using the QuEChERS Approach Combined with Both Liquid and Gas Chromatography**
Julie Kowalski, et al.; **Restek Corporation**, Bellefonte, PA

P-49a **Evaluation of Various Approaches for the Estimation of Measurement Uncertainty in Multi-Residue Methods for Fruit and Vegetables**
A.R. Fernández-Alba; **European Reference Laboratory EURL**, U. of Almeria, Spain

P-49b **Pesticide Residue Analyses of Fruit Juices by Direct Injection in LC-MS/MS Minimizing Matrix Effects**
Amadeo Fernandez-Alba; **European Reference Laboratory EURL**, U. of Almeria, Spain

P-50 **Update on Proposed and Approved ASTM Methods for Liquid Chromatography/Mass Spectrometry of Interest to US EPA and the Revival of the LC/MS Library Project**
Lawrence B. Zintek; **US EPA Chicago Regional Lab**, Chicago, IL

P-51 **Grain and Grain Dust Matrix Matching: Benefits and Pitfalls**
Robert D. Trengove, et al.; **Murdoch University**, Australia

P-52 **Optimization of HPLC/API-MS/MS Analytical Conditions in order to Reduce Matrix Interferences in the Simultaneous Detection of more than 30 Mycotoxins in Cereal Grains**
Robert D. Voyksner and Jennifer Sealey-Voyksner, **LCMS Limited**, Raleigh, NC

P-53 **State of Texas Approval for Organochlorine and Organophosphorus Pesticides by USEPA Method 8270 Full Scan LVI-GCMS**
Larry D. Garretson, et al.; **Scientific Instruments**, Round Rock, TX

P-54 **Application of Chemical Ionization GC/MS/MS for Trace Analysis of Pesticides in Foods**
Philip L. Wylie and Melissa Chruley; **Agilent Technologies**, Wilmington, DE

P-55 **Rapid Multi-Residue Analysis of Pesticides in Honey using a Buffered Acetonitrile Extraction and Dispersive Clean-Up**
Jonathan V Pelt, et al.; **Florida Dept. of Agriculture and Consumer Services**, Tallahassee, FL

P-56 **Comparison of Matrix Effects on QuEChERS and Dual-Layer SPE Method of 70 Pesticides from Six Agricultural Products using Triple-Quadrupole Mass Spectrometer**
Yu-Ri Lee, et al.; **NAQS, MIFAFF**, Chung-Nam Province, Republic of Korea

P-57 **Characteristics of Pesticide Residues and Dissipation in Mushrooms During Growth**
Guk-Tak Han, et al.; **NAQS, MIFAFF**, Chung-Nam Province, Republic of Korea

P-58a **Identification of Pesticide Residues in Essential Oils by QuEChERS Extraction and GC/MS Analysis**
Joan M. Stevens; **Agilent Technologies**, Wilmington, DE

P-58b **GC-µECD Analysis and Confirmation of CLP Pesticides in Olive Oil**
Joan M. Stevens; **Agilent Technologies**, Wilmington, DE

P-59a **Increasing Efficiency and Pesticide Recovery from the QuEChERS Approach for Fruits and Vegetable Products using Ceramic Homogenizers**
Joan M. Stevens; **Agilent Technologies**, Wilmington, DE

P-59b **Determination of Quinolone Antibiotics in Bovine Liver using QuEChERS Extraction with Analysis by LC/MS/MS**
Joan M. Stevens; **Agilent Technologies**, Wilmington, DE

P-60 **LC-APPI-MS/MS for Trace Analysis of "Difficult" Pesticides**
Sheng-Suan (Victor) Cai, et al.; **Syagen Technology**, Tustin, CA

P-61 **APPI Enhanced DART Source for Ambient Screening**
Jack A. Syage and Kaveh Jorabchi; **Syagen Technology**, Tustin, CA

P-62 **Pesticide Multiresidue Analysis in Tea Samples using Modified QuEChERS Method Combined with GC-NCIMS and LC-MS/MS**
Xian Zhang, et al.; **The Coca-Cola Company**, Atlanta, GA

P-63 **Accelerating Analytical Sample Preparation: Faster and Better by Flexible and Modular Automated Solutions**
Jon Cartlidge and Michael Schneider; **Chemspeed Technologies**, North Brunswick, NJ

P-64 **Pesticide Levels in Wines from the Niagara Region of Canada**
Sue C. Quade; **Health Canada**, Ottawa, Canada

P-65 **Evaluation of a QUECHERS Type Extraction and Cleanup for Tandem LC-MS Determination of Veterinary Drugs and Other Contaminants in Tissue Samples**
Michael S. Young, et al.; **Waters Corporation**, Milford, MA

P-66a **Determination of Pesticides in Wine by GC-MS: ITD-EI-MS, SQ-NCI-SIM and TQ-EI-MS/MS**
A. de Kok, et al.; **Food and Consumer Product Safety Authority (VWA)**, The Netherlands

P-66b **Simultaneous Determination of Pesticides and Mycotoxins in Wine by UPLC-MS/MS (ESI+)**
I.R. Pizzutti, et al.; **Federal University of Santa Maria**, Brazil

P-67a **Application of GC-MS (ITD-EI and SQ-NCI-SIM) and GC-MS/MS (TQ-EI) for the Development and Validation of a Multi-Residue Method for Pesticide Analysis in Breakfast Cereals**
A. de Kok, et al.; **Food and Consumer Product Safety Authority (VWA)**, The Netherlands

P-67b **Application of UPLC-MS/MS (ESI+) for the Development and Validation of a Multi-Residue Method for Pesticide and Mycotoxin Analysis in Breakfast Cereals**
I.R. Pizzutti, et al.; **Federal University of Santa Maria**, Brazil

P-68a **Multi-Method Development and Validation for Pesticide Analysis in Green Coffee Beans using GC-MS/MS (TQ-EI), GC-MS (SQ-NCI-SIM and ITD-EI)**
A. de Kok, et al.; **Food and Consumer Product Safety Authority (VWA)**, The Netherlands

P-68b **Multi-Method Development and Validation for 134 Pesticides and 36 Mycotoxins Analysis in Green Coffee Beans using UPLC-MS/MS (ESI+)**
I.R. Pizzutti, et al.; **Federal University of Santa Maria**, Brazil

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ORAL PRESENTATION ABSTRACTS

O-1 Multiclass, Multiresidue Analysis of Pesticides Typically Analyzed by Single Analyte Methods

Michelangelo Anastassiades, Diana I. Kolberg, and Dorothea Mack; **CVUA Stuttgart**, EU-Reference Laboratory for Pesticide Analysis by Single Residue Methods (EURL-SRM), Fellbach, BW, DE-70736, **Germany**; 0049-711-3426-1124, michelangelo.anastassiades@cvuas.bwl.de

Available multi-residue methods (MRMs) do not cover the entire range of pesticide/commodity-combinations. Non MRM-amenable pesticides require so-called single residue methods (SRMs) that focus on a single analyte or a group of analytes with similar characteristics (e.g. same moiety). Such methods typically have a less favorable cost-benefit ratio compared to MRMs, so labs, if at all, employ them very judiciously. Recent innovations in instrumental analysis opened the door for a straightforward analysis of numerous "difficult" pesticides and for the introduction of simple and broad-scope sample preparation procedures (e.g. QuEChERS). Nevertheless, still many pesticides are not covered by existing MRMs, requiring special conditions in sample preparation and/or instrumental analysis e.g. because of high lability, polarity and/or volatility or the need for cleavage to release bound residues or moieties. Within the frame of our EURL-SRM activities we focused on the analysis highly polar pesticides widely used in agriculture but still not monitored adequately. The aim was to develop a simple and readily transferable method, entailing a single extraction step and as few LC-MS/MS runs as possible. Missing isotopically labeled standards, the availability of which is essential for simplifying analysis, had to be ordered. The developed method has been applied on a wide range of market samples resulting in numerous findings.

O-2 Multiclass, Multiresidue Analysis of Pesticides, Veterinary Drugs, Environmental Contaminants, and Mycotoxins

Hans G.J. Mol, Paul Zomer, Theo de Rijk, Martijn van der Lee, and Arjen Gerssen; **RIKILT Institute of Food Safety**, Residue and Contaminant Analysis, Akkermaalsbos 2, Wageningen, 6708 WB, **The Netherlands**; +31 317 480318, hans.mol@wur.nl

In pesticide residue analysis, the use of multi-analyte methods is well-established and today methods covering hundreds of target analytes are commonly applied. More recently, a similar trend can be seen in other food-toxicant domains (mycotoxins, veterinary drugs, environmental contaminants). In all cases, methods for these contaminants are based on chromatography with mass spectrometric detection. An obvious continuation of the current trend is to integrate multi-analyte methods from different groups of contaminants into even more generic and comprehensive (screening) methods. Such methods are faster, provide more information and greatly reduce costs, especially in terms of $/analyte. In this presentation different options for generic extraction are compared for combined determination of various types of contaminants in a variety of food and feed matrices. The extracts obtained are highly complex and potentially contain any of many hundreds of toxicants. Simultaneous measurement of such large numbers of analytes is complicated with targeted acquisition methods such as SIM or MS/MS and, therefore, full scan MS detection is an attractive alternative. Different options will be discussed. Experiences with generic full scan methods will be presented, including data handling challenges.

O-3 How to Devise a "Greener" Lab and Facility

Jonathan Friedan; **Ballinger**, 833 Chestnut St., Suite 1400, Philadelphia, PA 19107; (215)446-0700, jfriedan@ballinger-ae.com

The environmental impact of laboratory facilities (and many other human activities) is unavoidable, but the reduction of that impact to a minimum is a worthwhile goal. It is in keeping with the mission of chemical monitoring programs whose facilities may ironically be having negative impacts. This presentation will provide examples of how to conserve resources, recommission and/or renovate your labs/facilities to improve energy efficiency, save water, and thus lower costs as well as reduce the environmental impact of your lab operations while, in many cases, improving the research environment.

O-4 Controlling Hormonal Residues in Animal Biological Matrices: from Conventional Targeted Methods to Emerging Global 'Omic Approaches

Jean-Philippe Antignac; **ONIRIS, LABERCA**, Route de Gachet, BP 50507, Nantes, Pays de la Loire, 44307, France, 33 240 68 78 80, jean-philippe.antignac@oniris-nantes.fr

A first aim of this lecture will be to present the current state-of-the-art regarding typical targeted approaches used in the field, including conventional and well mastered MS/MS methods on triple quadrupole instruments (QqQ) focusing on parent compounds or phase I/II metabolites. A second objective will be to present also more emerging approaches consisting to the non targeted measurement of a wide panel of analytes through high resolution full scan acquisitions using time of flight (TOF) or Orbitrap devices. Advantages and limits of these two strategies will be discussed, before introducing a third possible way for current laboratories, namely the 'omic approaches. This last aspect will be limited to metabolomic or sub-metabolomic strategies, through the presentation of three distinct real case studies for which first results were already published [1-3]. These illustrating examples are referring to differential (comparative) metabolomic studies using high resolution MS fingerprinting on TOF or Orbitrap instruments, which were dedicated

to (1) equine recombinant somatotropin, (2) beta-agonists in bovine (including a comment on a combined multi-level metabolomic/ proteomic/transcriptomic approach), and (3) steroid hormones assessed by several and complementary methods (GC-MS, GCxGC-HRMS, LC-HRMS). Our conclusion will discuss and balances all these approaches in terms of performances and compatibility toward the current regulation.

O-5 Analysis of antiviral drug residues in poultry and swine by LC-MS/MS

Shani Smith, Chaitali Chattopadhaya, Michelle Smith, Badar Shaikh, Jeffrey Ward, Mary C. Carson, Danny Chan[2], Jonathan A. Tarbin[2], and Matthew Sharman[2]; **U.S. Food & Drug Administration**, Center for Veterinary Medicine, Office of Research, 8401 Muirkirk Rd, Laurel, MD 20708; (301)210-4652, mary.carson@fda.hhs.gov
2. Food and Environmental Research Agency, Chemical Residues, Sand Hutton, York, YO41 1LZ, **UK**

In November 2005, the WHO, FAO and OIE jointly urged their Member States to not use antivirals in animals, in order to preserve their efficacy for human use. The U.S. FDA prohibited the extralabel use of adamantanes and neuraminidase inhibitors in chickens, turkeys, and ducks in early 2006. We report here a collaborative effort between CVM and FERA on the development of an LC-MS/MS method to enforce these prohibitions. The method target level is in the low µg kg^{-1} range. The analytes of interest are amantadine, rimantadine, oseltamivir phosphate, its active metabolite oseltamivir carboxylate, and zanamivir. These compounds have diverse chemistries, with the last two being highly hydrophilic zwitterions. Acidified water and acetonitrile (ACN) effectively extracts all five analytes from tissue. Several chromatographic approaches were evaluated. The best results were obtained using the ZIC silica-based HILIC column with an ACN:aqueous gradient. Two or more product ions were monitored for each analyte, allowing confirmation of identity. The method was validated with control, fortified, and incurred-residue tissues from chicken and turkey, and with control and fortified tissues from duck. When H1N1 flu emerged in 2008, the method was expanded to swine tissues.

O-6 Improving Analytical Efficiencies in Single-Analyte Methods for Veterinary Drug Residues in Foods of Animal Origin

Bryn Shurmer; **Canadian Food Inspection Agency**, Centre for Veterinary Drug Residues, 116 Veterinary Rd., Saskatoon, SK S7N 2R3, **Canada**; (306)975-5351, Bryn.Shurmer@inspection.gc.ca

In recent years, there has been a definite trend in veterinary drug residue testing towards mass spectrometry-based multi-residue methods. However, for various reasons, many labs still run more traditional single-class methods based on less selective detectors. The CFIA Saskatoon Lab has several motivations for maintaining capabilities in these types of single-analyte methods. We provide technical auditors to assist the Standards Council of Canada oversee accredited labs and need to maintain expertise on a wide array of analytical tools used to determine veterinary drug residues. Also, as a regulatory agency with international ties, the CFIA is interested in supporting methods and technologies that are accessible to developing countries. The use of traditional methods does not limit the opportunities to improve analytical efficiencies. In this work, short (1 to 2 cm), cost-effective “mini-columns” were used to improve chromatography in traditional single-analyte or single-class methods, resulting in lower run-times, higher through-put and in some cases, lower detection limits.

O-7 What You Always Wanted to Know About Root Cause Analysis and Managements Review, but Were Afraid to Ask

Arlene R. Fox, Senior Director; **AOAC International**, Proficiency Testing, 481 N Frederick Ave, Gaithersburg, MD 20877; (301)924-7077, afox@aoac.org

Two major challenges of achieving accreditation are root cause analysis and management review. These two requirements can be a source of great difficulty and pain or they can be great tools for solving problems and continuous improvement. Many laboratories have sought solutions before investigating the root cause of the problem. Learn how you can determine the root cause and prove that the corrective action worked and know the cause of that nonconformance has been eliminated. Problems that are not resolved consume enormous amounts of laboratory resources. No one enjoys fixing the same nonconformance over and over. Gain knowledge of how to benefit more from management review meetings. Many laboratories that are new to accreditation do not have experience with management review. The goal of their first attempt to conduct such a meeting is frequently to be able to mark off a checklist requirement. Management review meetings are an opportunity to evaluate the suitability and effectiveness of the management system. Learn how to increase your benefit from management review meetings.

O-8 ISO 17025, Section 5.9, or How I Learned to Stop Worrying and Love the Bomb

Gail A. Parker, Quality Manager; **FL Dept Agriculture and Consumer Services**, Chemical Residue Lab, 3125 Conner Blvd, L29 Bldg 3, Tallahassee, FL 32399-1650; (850)617-7513, parkerg@doacs.state.fl.us

The requirements of ISO 17025, Section 5.9 (Ensuring the Quality of Test Results), are brief and general, allowing for wide latitude in a laboratory’s implementation, but little guidance. AOAC International, via their ALACC document, has added several pages of

guidance to this section of 17025, but much of it is more applicable to single-parameter analyses than to multi-residue methods. Our typical QC samples include spikes of all the compounds analyzed in a given batch, resulting in over 22,000 individual recovery data points per year. In preparing for, attaining, and maintaining our laboratory's 17025 accreditation, we encountered, and continue to encounter, challenges in interpreting and in implementing the accreditation requirements in a way that adds real value without triggering unproductive corrective actions. This presentation will discuss some of our approaches, failures, and successes in managing a large volume of recovery data with respect to QC acceptance criteria and trend analysis. Discussion will also include how proficiency test (PT) sample results have been used to evaluate our methods and respond to non-conformances.

O-9 Statistics Without Math

Barry D. Nussbaum; **US Environmental Protection Agency**, Office of Environmental Information, 1200 Pennsylvania Ave., NW, (2842T), Washington, DC 20460; (202)566-1493, nussbaum.barry@epa.gov

This is a presentation of environmental statistics that gives tips on viewing data to find statistical conclusions without mathematics and formulas. There are two parts to the brief presentation. First, environmental statistics are presented in perspective to other published governmental statistics. Illustrations are provided in which "official statistics" measuring the same things seem to differ greatly and inconsistently. Second, concepts of statistical analysis and statistical sampling are presented in which illustrative techniques are employed in place of mathematics and formulas. The techniques that are presented really do corroborate Yogi Berra's famous observation that "You can observe a lot just by watching." While it was doubtful that Yogi was referring to mathematical statistics, his statement holds and conclusions from visual techniques will be highlighted. This session precedes the door prize drawing; so attendees have a chance to see probability theory in action, again without math.

O-10 Recent Developments in the European Pesticide Registration and Impact on Global Trade

Monika Bross; **BASF SE, Agricultural Center**, Crop Protection, Consumer Safety, 67117; Limburgerhof, **Germany**; +49 621 60-27139, monika.bross@basf.com

The registration of active ingredients for crop protection products in the European Union is a long process, requiring a large number of different studies and assessments. In the past years two important decisions were taken in Europe with regard to pesticide registrations. Both of them have or will have an impact on registrations in other countries and finally on global trade of treated commodities. In 2005, the EU MRL Regulation 396/2005 was introduced; it became effective on September 1, 2008. EEC 396/2005 has the goal to harmonize all MRLs / tolerances within Europe; it gives a very high priority to consumer safety and, consequently to dietary risk assessment. In the regulation, the ALARA principle for MRLs is mentioned. For non-target crops and for actives being not registered in Europe, "default" MRLs are set. It also includes several annexes which are under permanent revision and update. In 2009, the European Parliament voted for the Registration Regulation 1107/2009 ("revision of 91/414"); it will become effective in June 2011. The new regulation includes several points as e.g. hazard-based cut-off criteria, zonal authorisations and conditions for comparative assessment. In the presentation, an overview on the recent developments is provided; the consequences of the EEC 396/2005 are discussed in detail. Main focus will be given to the impact on residue analytical methods.

O-11 Challenges for Governmental Pesticide Laboratories in Brazil/Latin America

Ionara Regina Pizzutti; **Federal University of Santa Maria**, Av. Roraima 1000-Campus-Camobi Bldg. 18-Rm 2213, Santa Maria, RS, 97105-900, **Brazil**; 55 55 3220 9458, pizzutti@quimica.ufsm.br

Unlike colonization in North America, the general goal of Latin American colonization was to find and exploit natural resources rather than to construct a new homeland. This led to undesirable consequences and still has influences today in the way that Latin American countries are governed. Considering the natural resources and favorable climate conditions, Latin American countries are not poor, but the corruption and lack of administrative skills have largely kept the people from achieving a higher quality of life. These problems have also affected science and technological development in many ways, such as the process to establish a chain of qualified monitoring laboratories. In recent times, pressure from food importers necessitated actions to improve food safety and quality control of exporters. Nevertheless, there are still Latin American countries that do not have their own legislation and MRLs established, for instance. Over 20 years, Brazil has better established legislation to set MRLs for many residues/contaminants in agricultural products, and now a huge effort is being conducted by the Ministries of Agriculture (MAPA) and Science and Technology (MCT) toward constructing a network of laboratories, which will remain independent of changes in government. CEPARC/UFSM was invited to participate in this network, which is made up of 14 Brazilian labs, and an additional 6 labs in MAPA. Pesticide monitoring programs are being performed by the Ministry of Health using some labs of this network. The positive influence of these developments on the CEPARC/UFSM laboratory will be shown.

O-12 Pre-Harvest Monitoring and Management of Pesticide Residues in India

Kaushik Banerjee and Pandurang G. Adsule; **National Research Centre for Grapes**, National Referral Laboratory on Pesticide Residues in Fruits and Vegetables, P.O. Manjri Farm, Pune, Maharashtra, 412 307, **India**; 91-20-2691 4245, kbgrape@yahoo.com

In India, pesticide residue monitoring in fruits and vegetables started in 2003-04. It integrates all aspects of good agricultural practices and all the stakeholders. Government, farmers, exporters, pack-houses, residue testing laboratories, quality and phytosanitary certification authorities are adequately coordinated under the leadership of the National Referral Laboratory (NRL) and APEDA, Ministry of Commerce, Government of India to establish complete traceability of food safety through a unique web-based pre-harvest residue monitoring system. The whole process of residue management starts with farm registration (1-1.2 ha area) through a unique twelve digit alphanumerical code. A list of pesticides is recommended on the basis of the results of supervised multi-location field experiments. The dose, application instructions and pre-harvest interval of each pesticide are recommended to the farmers and their compliance is strictly monitored to regulate safe usage of pesticides at the farm level and minimize the residues at harvest. Currently 99 chemicals are monitored in each grape consignment, which includes 46 recommended and 53 others that might appear in grapes from indirect sources or are designated as banned pesticides. Random sampling is done from fields through a standardized procedure and analyzed by accredited laboratories (ISO/IEC 17025) by GC-MS and LC-MS. Performance of the laboratories is monitored by the NRL through inspection and proficiency testing rounds conducted at regular intervals. The program has significantly improved the export of Indian grapes with reduction in non-compliance to EU-MRL **from 24% to 3-3.5%** over last seven years.

O-13 Monitoring of Pesticide Residues in Export Products of Plant Origin in China

Jian-Hua Wang; **Shandong Exit & Entry Inspection and Quarantine Bureau**, No.70, Qutangxia Road, Shandong, Qindao, 266002, **China**; whywrs@yahoo.com.cn

In April 2001, the State Council of the People's Republic of China established the General Administration of Quality Supervision Inspection and Quarantine (AQSIQ) organization from 3 former competent authorities. AQSIQ is responsible for ensuring the food safety of exports. AQSIQ oversees 35 provincial level Entry-Exit Inspection and Quarantine Bureaus (CIQs), with almost 300 main branch offices and 200 local offices nationwide, with more than 6000 professionals who are directly involved in inspection of imported and exported food. Laboratory tested involves 163 food inspection and quarantine technical centers, and more than 1200 professionals. All of the laboratories have achieved China Metrology Accreditation (CMA) from the Certification and Accreditation Administration of China (CNCA) and additionally accredited to ISO 17025 by the China National Accreditation Service for Conformity Assessment (CNAS). All laboratories have participated in a substantial number of proficiency tests for pesticide residues in produce organized by CNCA, CNAS and international suppliers, such as the Food Analysis Performance Scheme (FAPAS). In September of 2004, AQSIQ began implementing the export plant-origin food residue monitoring plan, which is aimed at providing early warning information to competent authorities for the control of pesticide and other substances in produce, thereby better assuring quality and safety of exported plant-origin foods. Exported produce from Shandong province accounts for >30% of Chinese exports in that category each year.

O-14 Pesticide Residue Analysis: Fifty Years of Accomplishments and Continuing Challenges

James N. Seiber; **University of California, Davis**, Department of Food Science and Technology, Robert Mondavi Institute, Davis, CA 95616; (530)752-2490, jnseiber@ucdavis.edu

Pesticide residue chemists have made remarkable progress in applying analytical techniques to the science and art of residue chemistry. Major contributions have been made in addressing residue-related challenges with foods, worker safety, and environmental transport and fate. This effort has continued the availability of needed crop and animal protection agents that are both safe and effective. Continuing this system of surveillance while recognizing that, once a chemical is released to a field, backyard garden, feedlot, or forest, finite residues will persist, poses continuing challenges. We are also challenged by the presence of non-pesticide toxicants in food and the environment. Residue chemists, and the ultrasensitive techniques they have developed, have been critical in dealing with adulterants, products formed during processing, and authenticity issues which go beyond the traditional scope of pesticide residue analysis. Past accomplishments and continuing challenges will be addressed from the perspective of recognizing the importance of this field of science to maintaining the quality and safety of our food supply and environment.

O-15 Measuring Environmental Exposures: The Other Key to Risk Assessment

Keith R. Solomon; **University of Guelph**, Centre for Toxicology, School of Environmental Sciences, Bovey Building, Guelph, ON, **Canada**; (519)824-4120 x 58792, ksolomon@uoguelph.ca

Risk assessment (RA) for agrochemicals is a process of assigning magnitudes, probabilities, and relevance to the adverse effects that may result from use of these products and is critical to making the correct decisions about registration. RA requires data on effects and exposures and the latter is the province of the residue analyst. Residue values are compared to concentrations that cause adverse

effects in a tiered process. In lower tiers, simple ratios or quotients of single exposure and effects values and may be used to express hazard or relative safety. Normally, the toxicity value for the most sensitive organism or group of organisms is compared to the greatest exposure. This may be made more conservative by division of the quotient by an uncertainty factor. Higher tiers of RA make use of probabilistic techniques to address variance and uncertainty in both the exposure and the toxicity data, thus explicitly addressing uncertainty in the data rather than using simple uncertainty factors. Probabilistic techniques are more commonly used in environmental RA but have been applied to the characterizing of exposures for RA for the protection of humans. This presentation will address the role of the analyst and analytical data in the process of RA.

O-16 Helping the Farmer Helps the Regulator, Retailer, Consumer, and Environment: Everybody Wins

Yitzchak Skalsky, Ruty Spier, and Danit Klein; **Lab-Path Ltd.**, 17 Hacharash, Nes Ziona, 74031 **Israel**; +972526215982, danitklein@gmail.com

Lab-Path is a leading Israeli food safety company which specializes in creating and implementing food safety programs for domestic and international manufacturers. By implementing quality assurance and food safety procedures, we attempt that produce will comply with any and all regulations specified, including pesticide residues, microbial contamination and heavy metals. Lab-Path has developed a work module which connects the "missing links": taking into account all sources of risk in the field, performing adequate testing programs, processing all information gathered while updating and integrating the various standards and overseeing the implementation of corrective actions. Lab-Path also issues a "Stamp of Approval" through which food consumers can receive all information about the purchased foods via our website WWW.LAB-PATH.CO.IL/ENG. Lab Path works exclusively with analytical laboratories which are ISO 17025 compliant, FAPAS Ring Test approved and accredited and approved by the PPIS. In 2004, when Lab Path began its work in the domestic market, **30%** pesticide irregularities were revealed. By 2008 the irregularities were cut down to **3.6%**. By creating and implementing quality assurance and food safety plans, pesticide irregularities have dropped from 30% to 3.6% within four years. By holding corrective actions we have managed to educate the manufacturers in the importance of food safety.

O-17 Ambient Desorption Mass Spectrometry: A New Challenge in Rapid Pesticide Residues / Other Chemicals Control?

Jana Hajslova, Lukas Vaclavik, and Tomas Cajka; **Institute of Chemical Technology**, Dept. of Food Chemistry and Analysis, Technicka 3, Prague, 16628, **Czech Republic**; 00420220443184, jana.hajslova@vscht.cz

Around 30 ambient ionization methods for mass spectrometry have been reported over the last six years. This new technique enables samples or objects high-throughput analysis in their native state in the open environment with no prior preparation. Currently, desorption electrospray ionization (DESI) and direct analysis in real time (DART) are undergoing the most rapid development in various fields including pesticide residues and other contaminants control. While DESI, making use of pneumatically assisted charged jet, shares the key characteristics with typical ESI in terms of enabling the analysis of materials over a large mass range without significant fragmentation, DART, relying upon the formation of a (distal) plasma discharge in a heated helium gas stream, represents APCI-related technique, thus offering orthogonal ionization potential to DESI. In this presentation, several aspects of DART-MS use in pesticide residues/contaminants analysis will be presented: (i) Rapid control of calibration standards (stability tests, identification of breakdown products); (ii) Comparison of DART-TOFMS vs. DART-orbitrap MS; (iii) Performance characteristics achievable by DART-MS in the context of current QA/QC guidelines.

O-18 What's New with the Pesticide Residue Program in the FDA?

Chris A. Sack; **Food and Drug Administration**, Kansas City District Office, 11630 W 80th, Lenexa, KS 66214; (913)752-2182, chris.sack@fda.hhs.gov

The US Food and Drug Administration's pesticide residue program is rising to meet the challenges of protecting the American consumer. In the last year pesticide coverage was increased with the collaboration and implementation of a new LC-MS/MS procedure that is initially capable of screening approximately 200 analytes in 15 minutes. The current GC-MS SIM methods have been shortened and consolidated to analyze ~ 300 pesticides in less than 20 minutes. Full scan GC-MS with spectral deconvolution coupled with standardized MS spectra searches has expanded pesticide screening capacity by an additional 500 compounds. A modified QuEChERS procedure was collaborated for use in conjunction with both GC and LC determinations. The program administration has also undergone an exhaustive review, and a new high throughput business model encompassing the entire pesticide regulatory process from sample collection to compliance action has been devised; the agency is currently conducting pilot studies to evaluate the process. Looking to the future, a high resolution LC-MS determination of over 500 pesticides using orbitrap technology and a GC-MS/MS determination to replace the GC-MS SIM technology are both under development. And the agency is in the process of evaluating automated lab data management systems and laboratory information systems to streamline data processing from collection of samples to reporting of residue results.

O- 19 Multiresidue Analysis of Pesticides in Botanical Dietary Supplements, Herbals and Dried Botanicals by GC-MS and LC-MS Techniques

Jon W. Wong; **U.S. Food and Drug Administration**, Center for Food Safety and Applied Nutrition, 5100 Paint Branch Pkwy, HFS-706, College Park, MD 20740; (301)436-2172, jon.wong@fda.hhs.gov

Multiresidue methods have been developed for the analysis of pesticides in botanical dietary supplements using GC-MS/MS and LC-MS/MS. LOQs were determined at the 10-20 µg/kg levels, and recoveries and relative standard deviations were 70-120% and <20%, respectively, for most of the pesticides. These procedures allow for the effective screening of different pesticide types in botanicals at low concentrations. The goal of this work is to present effective and rugged strategies that would lead to efficient procedures for the multiresidue analysis of pesticides in a wide variety of botanical products.

O-20 Determination of 200 Pesticides in 24 Food Matrices by Liquid Chromatography-Tandem Mass Spectrometry

Kai Zhang, Jon W Wong, Paul Yang[2], and Michael Smoker[3]; **FDA Center for Food Safety and Applied Nutrition**, 5100 Paint Branch Pkwy, HFS-706, College Park, MD 20740; (301)436-2318, kai.zhang@fda.hhs.gov
2. Ontario Ministry of the Environment, Etobiocke, Ontario, M9P3V6, **Canada**
3. FDA Office of Regulatory Affairs, Lenexa, KS 66214

A multi-residue analysis method for the determination of 200 pesticides was developed and validated in 24 food matrices. Samples were prepared using QuEChERS procedures and analyzed by LC-MS/MS. The LOQs for the majority of the pesticides are less than 10 µg/kg. Recovery studies were performed by fortifying the food matrices at concentrations of 10, 25, 100 and 500 µg/kg. The resulting recoveries were between 70% - 120% for 80% of the pesticides at 10 and 25 µg/kg and 90% of the pesticides at 100 and 500 µg/kg with RSDs less than 20%. Lower (<70%) and higher (>120%) recoveries were most likely from complications of pesticide polarity, difficulty in ionization, stability, or matrix interference. Matrix effects in the 24 food matrices were determined using the slope ratio of matrix matched to solvent only calibration curves in the concentration range of 1.0–100 µg/L. Matrix effects were dependent on pesticides type and food matrix. Signal suppression, ranged from approximately 10-50% was observed in most of the pesticides in various food matrices. The method was used to analyze incurred samples containing various classes of pesticides at concentrations ranging from 1-500 µg/kg.

O-21 Changes in the FSIS Field and Lab Operations on Residue Analysis

Terry J. Dutko; **USDA Food Safety Inspection Service**, Office of Public Health Science, Midwestern Laboratory, 4300 Goodfellow Boulevard 105D, St. Louis, MO 63120; (314) 263-2686, terry.dutko@fsis.usda.gov

The Food Safety and Inspection Service (FSIS), the U.S. Department of Agriculture's public health regulatory agency, works with the Environmental Protection Agency (EPA) and the Department of Health and Human Services' Food and Drug Administration (FDA) to control veterinary drug, pesticide, and environmental contaminant residues in meat, poultry, and egg products. Residue control is a cooperative effort. EPA and FDA have statutory authority for establishing residue tolerances or action levels, and FSIS, through the National Residue Program (NRP), tests animal tissues and egg products to verify that tolerances or action levels are not violated. FSIS collects samples of meat, poultry, and egg products at federally inspected establishments and analyzes the samples at FSIS laboratories for chemical residues of veterinary drugs, pesticides, and environmental contaminants. Laboratory findings that exceed established tolerances or action levels are shared respectively with FDA and EPA. Since 1967, FSIS has administered the NRP to collect data on chemical residues in domestic and imported meat, poultry, and egg products. The NRP is designed to provide: (1) a structured process for identifying and evaluating compounds of concern by production class; (2) the capability to analyze for compounds of concern; (3) appropriate regulatory follow-up of reports of violative tissue residues; and (4) collection, statistical analysis, and reporting of the results of these activities. This presentation will describe the NRP process along with changes associated with the sampling and testing activities within FSIS.

O-22 20-Year Retrospective and Update of the Pesticide Data Program

Diana E. Haynes, Technical Director; **USDA, AMS, S&T**, Pesticide Data Program, 8609 Sudley Road, Suite 206, Manassas, VA 20110; (703)330-2300x134, diana.haynes@ams.usda.gov

The Pesticide Data Program (PDP) was initiated in 1991 to collect data on pesticide residues in food, with an emphasis on commodities highly consumed by infants and children. PDP data are used primarily by the U.S. Environmental Protection Agency to prepare realistic pesticide dietary exposures for continuing pesticide re-registration activities in accordance with the 1996 Food Quality Protection Act and ongoing registration review responsibilities. PDP is a Federal-State partnership; there are currently twelve cooperating sampling and testing States: California, Colorado, Florida, Maryland, Michigan, Minnesota, Montana, New York, Ohio, Texas, Washington, and Wisconsin. Together these States represent over 50% of the U.S. population. To date, PDP has tested 93 commodities and includes

over 340 pesticides and metabolites in its testing profiles. In 2010, PDP is testing apples, asparagus, bell peppers, cabbage, cantaloupe, cilantro, sweet corn, cucumbers, grapes, lettuce, mangoes, oranges, pears, sweet potatoes, watermelon, canned beans, canned/frozen spinach, oats, catfish, eggs, and drinking water (from municipal as well as groundwater sources) using multiresidue methods. A survey for macrocyclic lactones in lettuce is underway, and, later during 2010, PDP plans to add baby food to the program and to reintroduce soybeans or wheat.

O-23 EPA: Risk Assessment and Monitoring Data - Historical Aspects

Susan Hummel; **EPA Office of Chemical Safety and Pollution Prevention**, OPP Health Effects Division, 1200 Pennsylvania Ave., NW (7509P), Washington, DC 20460; (703)305-7689, hummel.susan@epa.gov

Risk Assessment is an important part of the pesticide review process at EPA. EPA reviews pesticide data supporting pesticide product registration, tolerance (MRL) setting, periodic reevaluation of registrations and tolerances. International cooperation among government agencies has increased. Pesticide Risk Assessment methods have been updated over the years at the EPA Office of Pesticide Programs and will be updated in the near term future. Dietary exposure, occupational exposure, residential exposure, aggregate exposure, cumulative exposure and risk assessments will be discussed. The types of data used for each type of risk assessment will be discussed, along with refinements in the risk assessments, emphasizing the use of monitoring data. Models used for risk assessment will be discussed. Updates in the multiresidue method data requirements and other data requirements will be discussed, along with emerging issues in pesticide risk assessment, and how to find information on EPA assessments of pesticides.

O-24 Targeted GC Pesticide Analysis at FDA: Transitioning to GC/MS/MS from GC/MS with Selected Ion Monitoring

Greg E. Mercer; **US Food and Drug Administration, Pacific Regional Laboratory Northwest**, 22201 23rd Dr SE, Bothell, WA 98021; (425)483-4755, greg.mercer@fda.hhs.gov

A primary objective for the FDA pesticide laboratories is to develop the capacity to analyze a much larger number of samples in a high-throughput setting. In addition to changes in extraction techniques, instrumental changes are in the works. Under investigation at FDA's Pacific Regional Laboratory Northwest (PRL-NW), one proposed change is a new GC/MS/MS detection procedure targeting ~230 pesticides that have been detected by GC on regulatory samples at FDA. Using this procedure, one injection with a 21 min runtime will replace the three injections and 45 min runtimes required for the existing SIM procedures which target ~350 pesticides. Targeted analysis based on historical analytical findings will also help streamline instrument calibration, standard preparation and data interpretation. This monitoring scheme would be augmented by additional, non-targeted detection techniques (i.e., element selective GC and/or full scan GC/MS). Any new or unusual pesticides identified with the non-targeted systems would eventually be incorporated into the current GC/MS/MS procedure. Preliminary work suggests that the increased sensitivity and multimode inlet might also be used to eliminate the need for any extensive sample concentration steps. Details on the development of this procedure, a comparison to SIM sample results and applicability to different extraction techniques will be discussed.

O-25 Practical Tools for GC-MS Analysis of Pesticides in Complex Matrices

Katerina Mastovska; **Covance Laboratories**, Nutritional Chemistry and Food Safety, 671 S Meridian Rd., Greenfield, IN 46140; (317)467-3645, katerina.mastovska@covance.com

When it comes to GC analysis of pesticides, both volatile and non-volatile matrix co-extractives can significantly affect method performance. To address some of the matrix-related issues in GC, multiple tools have been introduced that show promising results, but only a few have been implemented in routine laboratories. Why is that? A new tool has to be both cost-effective and easy-to-use to be considered for implementation. And it has to be rugged and reliable for daily operation. While GC-MS is the technique of choice for modern multiresidue analysis of GC-amenable pesticides, the selectivity of single-stage or nominal mass-accuracy MS may not be adequate to provide reliable quantitation and identification. After successfully implementing triple quadrupole LC-MS/MS, many laboratories are now considering triple quadrupole GC-MS/MS. While GC-MS/MS offers the same selectivity improvements, it can be only as successful as the weakest link in the entire system, which is often sample introduction. This talk will examine several GC-MS tools and techniques that, while not widely adopted, greatly aid pesticide residue analysis and meet the criteria for routine laboratory use. Practical techniques will be discussed that specifically deal with dirty matrices and help to make pesticide analysis far more rugged.

O-26 Development of a Sample Preparation Method and Analytical Assay using LC-MS/MS for Quantitation of 10 Mycotoxins in a Single Analysis

Lauryn E. Bailey, Curtis W. Jarrand, and Stephen P Powers[2]; **Eurofins Scientific**, 2315 N. Causeway Blvd, Suite 150, Metairie, LA 70001; (504)297-3422, laurynbailey@eurofinsus.com
2. Vicam, A Waters Business, Research and Development, Milford, MA, 01757

Mold-producing mycotoxins continue to be a growing agricultural concern in the production of corn and grain products for human and animal consumption. While regulatory agencies seek to enforce action limits on these toxins, sampling and analytical methods must also keep-up to accommodate these limits and reliably assess the toxin content in a variety of commodities. Traditional mycotoxin analyses require multiple different extractions, sample clean-ups, and analytical assays or chromatographic runs per toxin of interest – basically, multiple assays for a single sample. Here, we present work toward the development of a mycotoxin screen capable of detecting 10 major toxins (Aflatoxins B1, B2, G1, G2; Ochratoxin; T2 toxin; Vomitoxin; Zearalenone; Fumonisins B1, B2) in a single sample preparation using Vicam Myco 6-in-1 Immunoaffinity clean-up and a single chromatographic run using LC-MS/MS. While additional work is still needed, our data reveal significant steps forward in the development of a single sample preparation for a complete and accurate mycotoxin screen that achieves quantification limits below current action limits with the potential to be a suitable alternative to other mycotoxin analyses.

O-27 Flow Injection Mass Spectrometry for High-Throughput Pesticide Residues Measurements

Sergio C. Nanita, Anne M. Pentz, Lisa Swaim[2], and Bob Plastridge[2]; **DuPont Crop Protection**, Stine-Haskell Research Center, 1090 Elkton Road, S315/1220, Newark, DE 19714; (302)451-5806, sergio.c.nanita@usa.dupont.com
2. ABC Laboratories, Chemical Services, Columbia, MO 65202

The concept of applying flow injection mass spectrometry for fast residue analysis has been evaluated[1]. Flow injection is among few sample introduction techniques allowing mass spectrometric analysis without chromatography. The technique has been tested with HPLC/MS/MS instruments used in flow injection mode, i.e. bypassing the HPLC column compartment, providing tremendous improvement in analysis speed. The sample band injector-to-detector travel time was 6 seconds, and the sample bandwidth was 7 seconds. Analyte detection was followed by 10–45 seconds of solvent flush to rinse the injector-to-detector transfer line and ion source. This procedure allowed run times of 25–60 seconds, and has been tested for sulfonylurea herbicides, carbamate and anthranilic diamide insecticides, and various experimental active ingredients. Standards prepared in control matrix were necessary to compensate for matrix effects and achieve reliable calibration. Correlation coefficient (R^2) > 0.99, relative standard deviation (RSD) < 20%, and average percent recoveries between 70-120% have been obtained, demonstrating that flow injection MS/MS methods can meet regulatory guidelines. The advantages and limitations of the technique will be discussed. A method concept for even faster (qualitative) high-throughput pesticide residue screening will also be presented.
[1] Anal. Chem., 2009, 81, 3134–3142

O-28 Organochlorine Pesticides Determination in Strawberries and Jam using QuEChERS Extraction and GC-MS/MS

Valentina Fernandes Domingues[1],Virgínia Cruz Fernandes[1,2], Nuno Mateus[2], and Cristina Fernandes Delerue-Matos[1]; 1. **Requimte – Instituto Superior de Engenharia do Porto**, Dep. Química, Rua Dr. António Bernardino de Almeida 431, 4200-072 Porto, **Portugal**, 00351-228340500, vfd@isep.ipp.pt
2. CIQ – Centro de Investigação em Química, Universidade do Porto, Porto, **Portugal**

Organochlorine pesticides (OCPs) are prevalent environmental contaminants with high lipophilicity and long half-lives. During the last decades, public concern over pesticide residues in fresh fruit has led to the development of alternative cultivation systems in order to minimize the negative effects of conventional cultivation to the environment and consumer health. The aim is to develop an analytical method to determine eleven OCPs in strawberries and jam obtained from organic and integrated pest management farming. The QuEChERS method has been readily accepted for many pesticide residues analysis in food so it was selected for strawberries and jams sample preparations. An additional step in procedure was added in sample preparation with jams. The proposed method was successfully utilized for determination of OCPs in strawberries and jams from integrated pest management and organic farming in Portugal. The average recoveries obtained for each pesticide in strawberries and jam ranged between 54-128%. The LOD (3.4-8.7µg L^{-1}) of these pesticides were much lower than the MRL, allowed in Europe. In this presentation the benefit of QuEChERS is demonstrated in the trace analysis of OCPs.
Acknowledgement: This research was supported by a Ph.D. grant from FCT(BD/47200/2008)

O-29 Pesticide Residue Analysis of Catfish for the USDA-Pesticide Data Program (PDP)

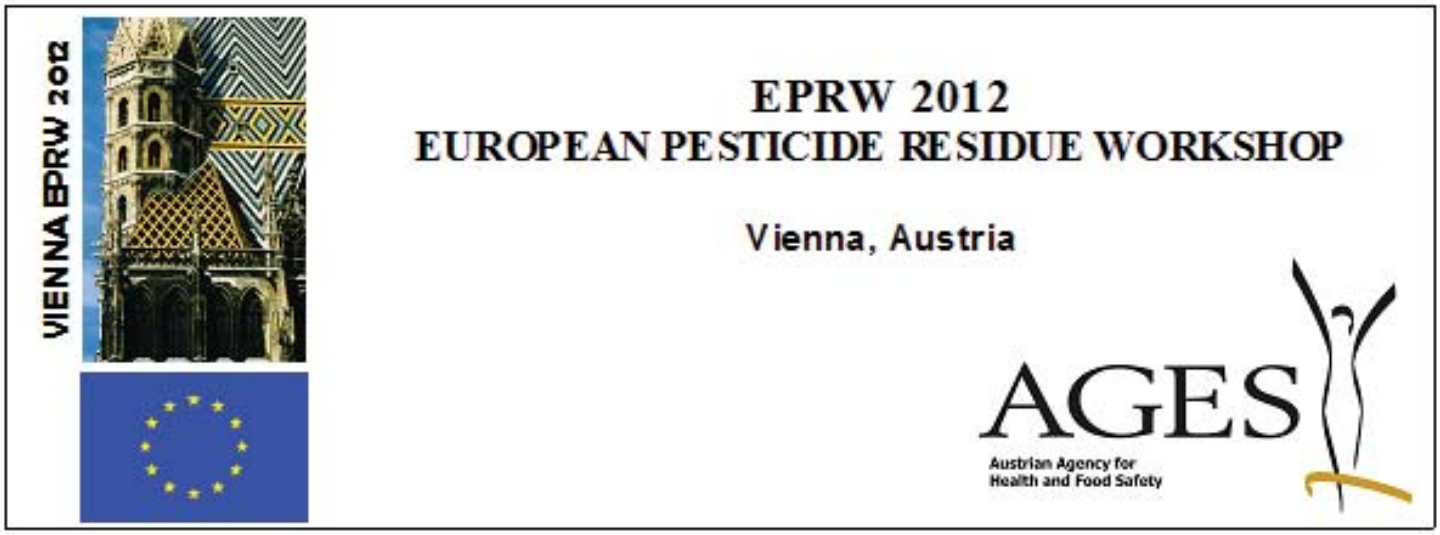
Jonathan Barber, and Roger Simonds; **USDA-AMS**, National Science Laboratory, 801 Summit Crossing Pl Ste B, Gastonia, NC 28054; (704)833-1523, jonathan.barber@ams.usda.gov

Samples of domestic and imported catfish, both farm-raised and wild, were collected and tested for pesticide residues from April1, 2008 – March 30, 2009 and from July 1, 2009 – June 30, 2010. Sixty-two samples per month were collected by PDP samplers at distribution centers and terminal markets under PDP's sampling frame, a statistically-based system that yields nationally representative samples of what is available to the consumer. Eligible samples included fish labeled as "catfish" that were fresh or frozen raw, in multiple forms, including fillets, nuggets, strips, or steaks, with or without bone. Pesticide residue analysis was performed by the USDA-AMS-National Science Laboratory in Gastonia, NC. Sample preparation, extraction methodology, and results will be discussed.

O-30 Determination of Three Neonicotinoids and their Metabolites in Pollen and Nectar of Field Treated Pumpkin to Assess their Potential Exposure to Honey Bees

Alaa Kamel, and Galen Dively[2]; **US EPA**, Office of Pesticide Programs, 701 Mapes Rd, Fort George G. Meade, MD 20755; (410)305-2925, kamel.alaa@epa.gov
2. University of Maryland, Department of Entomology, College Park, MD

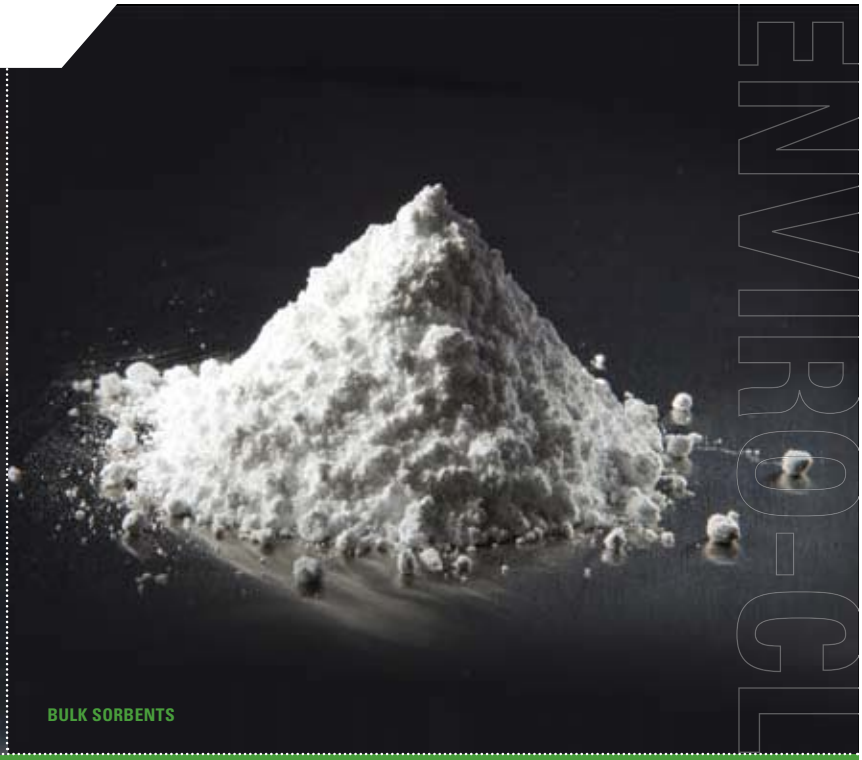
The magnitude of the residues of imidacloprid, dinotefuran and thiamethoxam as well as their metabolites was determined in pollen and nectar collected from pumpkin (Cucurbita pepo L.) fields treated at label-approved application rates to assess neonicotinoid exposure levels that may cause disorientation and associative learning problems in honey bees. Pollen and nectar were extracted with 2% triethyl amine in acetonitrile with the aid of a salting out agent and further cleaned by solid phase extraction (SPE) through C-18 cartridges. Qualitative and quantitative analysis was carried out by ultra high performance liquid chromatography – tandem quadrupole mass spectrometry (UPLC-MS/MS). Detected residues of applied neonicotinoids varied depending upon the type and timing of application, and were found in pollen at a relatively higher concentration than in nectar samples. Residues of imidacloprid ranged from 3 to 102 ppb in pollen and 0.3 to 13 ppb in nectar, residues of dinotefuran ranged from 36 to 147 ppb in pollen and 5.8 to 10.3 in nectar, and residues of thiamethoxam ranged from 55 to 127 ppb in pollen and 7 to 12.2 ppb in nectar. Residues of the metabolites of the three neonicotinoids were also found and ranged between 0.2 to 41 ppb in pollen and 0.3 to 10.8 ppb in nectar samples. Residues were highest for foliar and chemigation applications applied when flowers were present.





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POSTER ABSTRACTS

P-1 Dissipation of Dimethazone Residues in Agricultural Field

George F. Antonious; **Kentucky State University**, Plant and Soil Science, 400 East Main Street, 218 Atwood Research Center, Frankfort, KY 40601; (502)597-6005, george.antonious@kysu.edu

A field study was conducted to monitor off-site movement and persistence of dimethazone (an herbicide). Eighteen plots were separated using metal borders and the soil in six plots was mixed with sewage sludge and yard waste compost (SS+YW), six plots were mixed with SS, and six unamended plots (NM) were used for comparison purposes. The objectives of this investigation were to: i) monitor the dissipation of dimethazone in soil under three management practices; and ii) determine the concentration of dimethazone in runoff and infiltration water following natural rainfall events. GC/MS analyses of soil extracts indicated the presence of an ion fragment at *m/z* 125 that can be used for identification of dimethazone residues. Decline of dimethazone residues in the top 15 cm native soil and soil incorporated with amendments revealed half-life ($T_{1/2}$) values of 3.26, 2.01, and 8.05 d in SS+YW, SS, and NM treatments, respectively. Addition of SS+YW to native soil increased water infiltration, lowering surface runoff water volume and dimethazone residues in runoff following natural rainfall events.

P-2 LC-ESI-MSMS Analysis of Toxic Industrial Chemicals from UV/TiO2 Photocatalytic Oxidation

William C. Heiser, Irene C. Poyer, and Joel Murry; **Advanced Concepts and Technologies International**, 316 Kelly Drive, Waco, TX 76710; (254)751-9823, wheiser@act-i.com

This study incorporates the development of an Applied Biosystems MDS SCIEX 4000 QTrap™ LC-ESI-MSMS analytical method (MRM mode) with the evaluation of a UVλ_{254}/TiO2 slurry-based flow-through advanced oxidative process. The treatment process investigated the degradation efficacy of a mixture of three Toxic Industrial Chemicals: phosphamidon, nicotine and triethyl phosphate (TEP). The UV/TiO_2 treatment technology was evaluated using a lab-scale Photo-Cat L™ (Purifics ES Inc.) unit. Solutions were prepared in deionized water (DI, pH 6.6-7.6), laboratory prepared high total organic carbon (TOC) water (HCW, pH 6.3-7.6, TOC 11.2 mg/L), and water collected from Lake Waco, TX (LW, pH 7.8-8.5, TOC 5.4 mg/L). The average initial concentration (C_0) was 1.14×10-5 M phosphamidon, 2.10×10-5 M nicotine and 1.77×10^{-5} M TEP. Pseudo first order k_{obs} and $t_{1/2}$ results were as follows: phosphamidon in DI (0.034 sec-1, 20.7 sec), HCW (0.009 sec-1, 75.2 sec), LW (0.0051 sec-1, 143 sec), nicotine in DI (0.026 sec^{-1}, 28.0 sec), HCW (0.014 sec^{-1}, 50.0 sec), LW (0.0072 sec^{-1}, 96.7 sec), and TEP in DI (0.012 sec^{-1}, 60.2 sec), HCW (0.019 sec^{-1}, 37.0 sec), LW (none observed). Work in progress is focused on determining the transformation byproducts from the UV/TiO_2 photocatalysis of phosphamidon.
Unclassified: Dist A. Approved for public release.

P-3 Translocation of ^{14}C-Glyphosate Between Seedlings of Brachiaria brizantha and Coffee (Coffea arabica) and Citrus (Citrus limonia)

Paulo Alexandre de Toledo Alves, Valdemar Luiz Tornisielo, and Alberto Henrique Ricordi; **Centro de Energia Nuclear na Agricultura CENA/USP, Laboratório de Ecotoxicologia**, Avenida Centenário, 303, Piracicaba, São Paulo, 13416-000, **Brazil**; 0055 019 34294676, atpaulo@yahoo.com.br

Glyphosate is largely used as herbicide for several important agricultural crops. Chemicals applied through leaves may be liberated in the soil solution due to root exudation, a process that might negatively affect the physiology of non-target economical crops. The experiment was conducted part in a greenhouse and part in the installations of the Ecotoxicology Laboratory. Seedlings of Catuaí coffee variety and citrus (non-target plants) were grown for 75 days in individual pots, when the target weed (Brachiaria brizantha) was sown. Six weed plants were selected for radiolabeled glyphosate application thirty days after their sowing. Wood stocks were used to separate target and non-target plants during application, avoiding any drift risk. Forty-five microliters of a radiolabeled solution with a specific radioactivity of 608.35 Bq ml^{-1} were applied in the selected target plants. Twelve days after the radiolabeled glyphosate application, soil, leaves and root samples were collected and submitted for radioactivity analyses by LSC. Was observed that ^{14}C-glyphosate was translocated from target plants to non-target plants. A decreasing gradient of radioactivity were verified, which indicates the dilution effect of the Glyphosate between target and non-target plants.

P-4 LC-MS/MS Analysis of Triazine Pesticides in Drinking Water using a New Software for Streamlined Method Development

Charles Yang, Jonathan Beck, Dipankar Ghosh, Jamie Humphries,[1] Kristi Akervik, and Louis Maljers; **Thermo Fisher Scientific**, 355 River Oaks Pkwy, San Jose, CA; (925)628-0929, charles.yang@thermofisher.com
1. Thermo Fisher Scientific, Software, 2215 Grand Avenue Parkway, Austin, TX

A software program was developed with built-in workflows for streamlining routine analysis in environmental and food safety labs. The analysts can easily incorporate a LCMS methods database that can be customized to include unique compounds and commonly

encountered contaminants found in the environment. Mixtures of triazine compounds were spiked into drinking water samples ranging from 1 to 100pg/ml. 20mL of spiked water and blank samples were injected directly (with online preconcentration) were analyzed, allowing detection of low pg/mL (ppt) level concentrations. After an appropriate time, a multi port valve is switched to enable the load column to be back flushed onto the analytical column, where the compounds are separated prior to introduction into a triple quadrupole mass spectrometer. After all compounds are eluted, the valve is switched to starting position and the load column is cleaned with high organic phase and equilibrated, as is the analytical column. The compounds analyzed include: Ametryn, Atraton, Atrazine, Prometon, Prometryn, Secbumeton, Simetryn, Simazine, Terbutryn. All of the analytes in the mixture was linear over the range. In addition to the advantage of having an online sample preparation setup, the large injection volume allows for the pre-concentration of samples on the load column, and eluted onto the analytical column. The analytes show excellent peak shapes, which would not be attainable if the same injection volume was injected directly onto the analytical column. Also, significant time savings are realized versus offline SPE and concentration.

P-5 Large Injection Volumes and Online Pre-Concentration for the Analysis of Pesticides in Beverage Utilizing High Resolution Accurate Mass LCMS

Kristi Akervik, Charles Yang, and Jonathan Beck; **Thermo Fisher Scientific**, 355 River Oaks Parkway, San Jose, CA; (408)965-6392, kristi.akervik@thermofisher.com

An online sample pre-concentration method was developed using direct injections of 1 mL of Vitamin Water® samples spiked with 60 pesticides. Two HPLC columns are utilized, one as loading/pre-concentration column (Hypersil GOLDaQ 20X2.1 mm, 12µ) and the second an analytical column (Hypersil GOLDaQ 50X2.1 mm, 1.9µ), for analysis of the compounds in the 1 mL mixture of the spiked vitamin water (pesticide mixture from 1 to 100 pg/mL) and blanks. After an appropriate time, a multi port valve is switched to enable the loading column to be back flushed onto the analytical column where compounds are separated prior to introduction into an Orbitrap mass spectrometer. The compounds were easily identified using accurate mass (and required no tuning for transitions) and showed an increase in response compared to normal injection volumes (10 µL). Average increase of ~5x for 1 mL was seen thus increasing the detection limits. Significant improvements in productivity are realized by using direct online LCMS sample pre-concentration in conjunction with high resolution accurate mass instrumentation.

P-6 Qualitative Aspects in the Analysis of Pesticide Residues in Fruits and Vegetables using Fast, Low-Pressure GC/TOF

Steven J. Lehotay,[1] Urairat Koesukwiwat,[1,2] Henk van der Kamp,[3] Hans G.J. Mol,[3] and Natchanun Leepipatpiboon;[2] [1]**USDA Agricultural Research Service**, 600 East Mermaid Lane; Wyndmoor, PA 19038; (215)233-6433, steven.lehotay@ars.usda.gov
2. Chulalongkorn University; Bangkok, **Thailand**
3. RIKILT Institute of food Safety, Wageningen, **The Netherlands**

Quantitative method validation is a well-established process to demonstrate trueness and precision of the results with the method. However, an assessment of qualitative results is also an important need to estimate selectivity and devise identification criteria for chemical residues with the method. For multianalyte analysis, automatic instrument software is commonly used to identify the target analytes by comparison of their mass spectra against a database library. Especially at low residues levels in complex matrices, manual checking of results is typically needed to correct the peak assignments and integration errors, which is very time-consuming. Low-pressure gas chromatography− mass spectrometry (LP-GC/MS) has been demonstrated to increase the speed of analysis for GC-amenable residues in various foods and provide more advantages over the traditional GC-MS method. In this study, we used LP-GC/MS on a time-of-flight (TOF) instrument for the quantification and identification of 150 pesticides in strawberry, tomato, potato, orange, and lettuce samples. The LP-GC/TOF provided high sample throughput with <10 min analysis time. In a quantitative validation, acceptable performances were achieved with overall recoveries 70-120% and <20% RSD for >126 analytes in >180 sample extracts at 3 spiking levels. In a qualitative assessment, we analyzed in blind fashion 130 samples spiked randomly with different pesticides at different levels in the different matrices. We compared automated software evaluation with manual assessments in terms of false positive and negative results. The automated approach yielded a slightly higher rate of false results, but with shorter time for data analysis compared to the manual verification approach.

P-7a Performance Evaluation of LC-Single Stage Orbitrap-MS for Screening of Residues and Contaminants in Food and Feed

Paul Zomer, Frans Schoutsen,[1] and Hans Mol; **RIKILT, Institute of Food Safety**, Akkermaalsbos 2, Wageningen, 6708 WB, **The Netherlands**; +31 317 480318, hans.mol@wur.nl
1. Thermo Fischer Scientific, Breda, **The Netherlands**

With the introduction of a single stage Orbitrap-MS, ultra-high resolving power/high mass accuracy MS has become a realistic option for mainstream applications such as screening and determination of pesticides residues and other toxicants in food and feed. Recently, the merits of full scan measurement using a resolving power (RP) of 100,000 (FWHM) were investigated for this application area [1]. In this work the performance of a single stage Orbitrap MS (Exactive, Thermo Fisher Scientific) for full scan screening of residues is

evaluated. Different matrices (tomato, wheat, meat, egg, and a compound feed) were extracted using a generic extraction/dilution method [2], and spiked with >200 toxicants at 0.01-0.25 mg/kg. The extracts were analyzed using alternating scan events in the same run: one without fragmentation and one with all-ion-fragmentation in the so-called High energy Collision Dissociation cell of the instrument (RP= 50,000). Data handling/reporting was done using automated library-based detection and the number of false positives and false negatives obtained were evaluated.

[1] M. Kellmann, H. Muenster, P. Zomer, H.G.J. Mol, J. Am. Soc. Mass Spectrom. 20 **(2009)** 1464
[2] H.G. J. Mol, P. Plaza-Bolanõs, P. Zomer, T.C. de Rijk, A.A.M. Stolker, P.P. J. Mulder, Anal. Chem., 80 **(2008)** 9450–9459

P-7b Target Screening and Quantification Software for GCxGC-TOF-MS Data

Guido van der Weg, Martijn van der Lee, Arjen Lommen, and Hans Mol; [1] **RIKILT, Institute of Food Safety**, Akkermaalsbos 2, Wageningen, 6708 WB, **The Netherlands**; +31 317 480318, hans.mol@wur.nl

GCxGC-TOF-MS analysis is a very powerful technique for screening of toxicants in food and feed [1]. For efficient use in routine practice, however, automated library-based identification is required. The currently available proprietary software (Leco ChromaToF in this case) is not optimally suited for this purpose. In this work a new alternative data processing tool was developed for automated library-based identification and concentration estimation of target compounds after analysis by GCxGC-TOF-MS. Raw data files are converted into net.CDF format and then pre-processed using earlier developed MetAlign software [2]. Next, the resulting reduced data files are searched against a user or commercial NIST-compatible library and the tentatively identified compounds, including an indicative concentration, are reported in Excel format. The screening performance was tested by detection of pesticides and contaminants in different food samples (fruit-based baby food, wheat, fish oil) which were analyzed without and with fortification (356 compounds). The target compounds could be reliably identified at low levels (>90% at 10 ng/g level) with very low numbers of false positives.

[1] M.K. van der Lee, G. van der Weg, W.A. Traag, H.G.J. Mol, J. Chromatogr. A 1186 (2008) 325-339
[2] www.metalign.nl/UK/ (download and publications)

P-8a Development of Immunochemical Methods (ELISA and Immunosensor) for Determination of Paraquat Residues in Food Samples

Raul Garcia-Febrero,[1] Enrique Valera,[1] Alejandro Muriano,[1] J.-P. Salvador,[1] Diana Kolberg,[2] Richard Fussell,[3] Hans Mol, Francisco Sanchez-Baeza,[1] and Pilar Marco;[1] **RIKILT, Institute of Food Safety**, Akkermaalsbos 2, Wageningen, 6708 WB, **The Netherlands**; +31 317 480318, hans.mol@wur.nl
1. IQAC-CSIC, Applied Molecular Receptors group, Barcelona, **Spain**
2. CVUA, Stuttgart, **Germany**
3. Fera, York, **United Kingdom**

Paraquat is a broad-spectrum herbicide used for weed control in various crops, as defoliant (cotton, hops) and for destruction of potato haulms. Since 2007 it has been banned in the EU. Paraquat is not amenable to multi-residue methods and rapid screening assays for detection in crops are desirable to verify compliance with Good Agricultural Practices and for monitoring purposes. In this work, immunochemical methods to detect residues of paraquat in crops have been developed. Antibodies have been produced by immunizing White New Zealand rabbits with N-(4-carboxybut-1-yl)-N-methylbipyridilium acid hapten, coupled to horseshoe crab hemocyanin (HCH). The microplate-based ELISA (Enzyme Linked Immunosorbent Assay) detects paraquat down to 0.39±0.21 ug L^{-1} in buffer (PBS (25% MeOH)) with a very high selectivity. In cross reactivity tests using similar substances, only monoquat interferes with a cross-reactivity of 21%. Preliminary studies for the development of an electrochemical immunosensor have been performed, using graphite composite electrodes (GEC), magnetic m-particles, and HRP (horseradish peroxidise) labelled paraquat antibodies. They have shown that it is possible to reach LOD values below 8 mg/L.

This work was done as part of the EU project CONtaminants in Food and Feed: Inexpensive DEtectioN for Control of Exposure (CONffIDENCE,) (FP7-211326-CP) www.conffidence.eu/index.php

P-8b Direct Analysis of Dithiocarbamates by DESI-MS/MS

Paul Zomer, Hans Mol, Michel Nielen,[1] Tomas Cajka,[2] and Jana Hajslova;[2] **RIKILT, Institute of Food Safety**, Akkermaalsbos 2, Wageningen, 6708 WB, **The Netherlands**; +31 317 480318, hans.mol@wur.nl
1. Wageningen University, The Netherlands
2. Institute of Chemical Technology, **Czech Republic**

Dithiocarbamates (DTCs) are a widely used group of fungicides. Analysis of these compounds has always been a challenge. The most common way to determination DTCs is through conversion into CS_2 which is then measured spectrophotometrically or by GC. These methods do not provide information on the parent DTC present in the sample. The latter is required in certain cases since specific MRLs have been set for thiram, ziram and propineb (2007/57/EC). In the frame of an EU funded project [1], ambient mass spectrometry is being investigated as rapid screening tool for detection of DTCs directly from sample surfaces with no, or minimal, sample preparation.

This might be favorable for detection of dithiocarbamates since they are non-systemic and solubility and stability issues could be circumvented this way. In this work we present the possibilities and limitations of DESI (Desorption ElectroSpray Ionization) coupled to MS/MS. Thiram was used as the compound to optimize the system. Different factors which impact the analysis of the dithiocarbamates will be discussed.

[1] CONffIDENCE, *CONtaminants in Food and Feed: Inexpensive DEtectioN for Control of Exposure* is funded under the European 7th Framework program (FP7-211326-CP) www.conffidence.eu/index.php

P-9a Direct analysis of Dithiocarbamates by Ambient Mass Spectrometry Employing a Direct Analysis in Real Time (DART) Ion Source

Tomas Cajka, Jana Hajslova, Katerina Riddellova, Paul Zomer,[1] Hans Mol;[1] **Institute of Chemical Technology**, Technicka 3, Prague, **Czech Republic**, 16628; 420 220 443 142, Tomas.Cajka@vscht.cz
1. RIKILT - Institute of Food Safety, Wageningen University, Wageningen, **The Netherlands**

Dithicarbamates (DTCs) represent an important class of fungicides widely used in agriculture. Typically, DTC-residue analysis is based on hot-acid digestion and determination of formed CS2 by various techniques (*e.g.* spectrophotometry, HS-SPME-GC-MS). However, specific maximum residue limits are increasingly set by European legislation (2007/57/EC) for certain fungicides, to be determined as they are applied (*i.e.* thiram, ziram, and promineb). In our study, we attempted to optimize a fast and rapid method for determination of thiram and ziram in fruit based on QuEChERS extraction. The crude extract was immediately analyzed employing ambient MS with a direct analysis in real time (DART) ion source coupled to high-resolution TOFMS. The main advantages of DART-TOFMS involved the possibility of direct sample examination in the open atmosphere, minimal sample preparation requirements, and, remarkably high sample throughput. Under these conditions, the analysis time was reduced significantly (<8 min for sample preparation and <1 min for extract examination by DART), thus, less degradation of DTCs typically observed in the presence of plant matrix can be expected. The performance characteristics obtained by this newly developed method will be discussed.

This study was carried out within the project FP7-211326-CP (CONffIDENCE) supported by the EC through the 7th Framework Programme.

P-9b Comprehensive Two-Dimensional Gas Chromatography–Time-of-Flight Mass Spectrometry (GC×GC–TOFMS) for the Determination of Multiple Pesticide Residues in Baby Food

Tomas Cajka, Marketa Hakenova, Katerina Riddellova, and Jana Hajslova; **Institute of Chemical Technology**, Technicka 3, Prague, **Czech Republic**, 16628; 420 220 443 142, Tomas.Cajka@vscht.cz

In recent years, GC×GC has attained increasing attention for its outstanding separation potential and capability to solve demanding analytical tasks. For some complex food matrices, such as baby food, the requirements on method detection limits are very strict (1999/39/EC, 2003/13/EC) and the unambiguous confirmation of the pesticide presence based on mass spectrometric detection is required (SANCO/10684/2009). It has been demonstrated that the application of GC×GC brings distinct advantages such as enhanced separation of target pesticides from matrix co-extracts as well as their improved detectability. In this study, GC×GC–TOFMS was optimized in terms of selection of stationary phases (orthogonal vs. reversed-type) and GC oven temperature programs for the determination of pesticide residues (>100) with a broad range of physico-chemical properties in fruit-based baby food. For sample preparation, QuEChERS protocol was used. The performance characteristics obtained by this developed method will be discussed.

This study was carried out within the project MSM 6046137305 supported by the Ministry of Education, Youth and Sports of the Czech Republic.

P-10 Automated Purification Method for OCPs in Sediment using a SPE-GPC-VAP System

Zhi Fen Gan,[1] Xing Ru Zhao,[1] Shu Suan Liang,[2] T.K. Dobbs, J. Wiseman, and J. Salmons; **J2Scientific**, 1901 Pennsylvania Dr., Suite C, Columbia, MO 65202; (573)214-0472, tdobbs@j2scientific.com
1. State Environmental Protection Key Laboratory of Estuary and Coastal Environment, Chinese Research Academy of Environmental Sciences, Beijing, **China**
2. College of Chemistry and Environmental Science, Hebei University, Baoding, Hebei, **China**

An efficient automated method has been developed for separation and quantitation of organochlorine pesticides (OCPs) in sediment using the Preplinc® SPE-GPC-VAP purification system and subsequent analysis by gas chromatography-mass spectrometry (GC-MS). The SPE uses LC-Florisil as the sample clean-up cartridge. The GPC column (Biobeads SX-3) was used with ethyl acetate/cyclohexane (1:1 v/v) as the mobile phase with UV detection at 254 nm. The fraction containing the analytes of interest were collected and concentrated on the AccuVap. The observed detection limits ranged from 0.03 ng/g to 1.31 ng/g of OCPs in dried sediment. The capabilities of this method were demonstrated by using sediment samples obtained from five regional areas of Bohai Bay in Tianjin, China. The levels of total OCPs determined in sediment ranged from 1.5 ng/g to 36.4 ng/g dry weight. The highest concentration was found in Dagu Rivers, followed by Nanpai Rivers. The current method has been shown to be rapid, effective, and suitable for purification of residues of OCPs from sediment samples.

P-11 MSPD Determination of Pesticide Residues in Beeswax

Silvina Niell, Andres Pérez, Natalia Martinez, Veronica Hernandez, Leonidas Carrasco Letelier,[1] Horacio Heinzen, and Veronica Cesio; **Facultad de Química, Pharmacognosy & Natural Products**, Gral. Flores 2124, Montevideo, 11800, **Uruguay**; (5982)(9244068), cs@fq.edu.uy; silvinaniell@gmail.com
2. Instituto de Investigacion Agropecuaria(INIA), La estanzuela, La estanzuela, Colonia, **Uruguay**

Beeswax is a pharmaceutical and cosmetic commodity listed in the US and EU Pharmacopeias and a raw material for many daily house products like floor waxes. Also, beekeepers recycle beeswax when they establish a new hive. Beeswax is a very complex mixture of lipophillic compounds secreted by bees to built up the hive but many organic pollutants can dissolve in it. Acaricides come from beekeeping activities but other environmental contaminants are carried into the hive when bees fly around searching food. Curiously, little attention has been paid to the presence of harmful pesticides in beeswax, which is an excellent vehicle for the entrance of pesticides residues through human and pets´ skin and a pollution reservoir for a new hive. In the present communication we present a straightforward, miniaturized MSPD method for the determination of pesticides and acaricides in beeswax. The sample was dispersed on RPC18 silica gel, the pesticides were eluted using 20 mL of acetonitrile saturated in hexanes, and determined through gas chromatographic methods (GC-FPD, GC-ECD , GC/MS). Recoveries were 80-120% with RSDs of 5-15% for GC amenable agrochemicals showing for each pesticide a different matrix effect. The method is not only accurate and reproducible but also environmentally friendly.

P-12 Straightforward Determination of 40 Organophosphorous Pesticides in Raw Wool using Microwave Assisted Extraction and GC/FPD Analysis

María V. Cesio, Silvina Niell, Lucía Pareja, Gabriel González, Zisis Vryzas,[1] Euphemia Mourkidou,[1] and Horacio Heinzen; **Facultad de Química, Pharmacognosy & Natural Products**, Gral. Flores 2124, Montevideo, 11800, **Uruguay**; (5982)(9244068), cs@fq.edu.uy; silvinaniell@gmail.com
1. Aristotle University of Thessaloniki, Department of Agriculture, Thessaloniki, Macedonia, **Greece**

Uruguay is the fourth world exporter of raw and processed wool. In order to keep this position, the improvement of both, quantity and quality of wool production, is a major objective for the Uruguayan industry. This depends greatly on the sanitary condition of flocks. Ectoparasites, such as lice, scabies and flies are common pests for sheep, and can deteriorate the quality of the fleece. Pesticides are currently employed to control them. The International Wool Trade Organization (IWTO) rules the different trading aspects, particularly those focused on pesticide residues in wool. Despite the fact there is no official analytical protocol to analyze these residues, IWTO suggests some procedures. The most frequently applied methodology is a laborious process where pesticides and grease wool are extracted with petroleum ether in Soxhlet, the resulting wool wax is fractionated through GPC, the pesticide residues collected and GC analysed. In the present communication we present a straightforward procedure where selective determination of 40 organophosphorous pesticides was accomplished using acetonitrile with the optimum conditions of MAE and GC/FPD analysis. Average recoveries at different levels were between 70-120% with RSD < 20% and LODs from 0.01 to 0.1 mg/kg. The method was also validated analyzing real wool samples.

P-13 Multi-Mycotoxin Analyses, including Cyclopiazonic Acid, in Various Grains by Mini-Column LC-MS/MS

Perry A. Martos, Wade Thompson, and Gonzalo J Diaz;[1] **University of Guelph**, Laboratory Services, 95 Stone Rd. W., Guelph, Ontario, N1H8J7, **Canada**; (519)212-4359, pmartos@uoguelph.ca
1. Universidad Nacional de Colombia, Facultad de Medicina Veternaria y Zootecnia, Bogota, **Colombia**

Mycotoxins represent one of the single most toxic groups of compounds in the natural environment and have caused a wide range of illnesses and even death in humans and animals. Under the right conditions, mold and fungi contaminated products can produce one or more mycotoxins. The study of multiple mycotoxins and mycotoxin classes often requires the implementation of multiple methods. It is therefore advantageous to analyze a wide range of mycotoxin classes with one analytical routine. This work focuses on the development of a simplified multi-mycotoxin method to study the most toxicologically relevant mycotoxins in Canada: aflatoxins, sterigmatocystin, cyclopiazonic acid, tricothecenes, ochratoxin A, fumonisins, zearalonone, and ergot alkaloids. Cyclopiazonic acid has not been included in other published multi-mycotoxin methods, reasons for which are explained based on our research. A method has been developed and validated that can extract and provide analysis for 22 mycotoxins using liquid chromatography with a 0.75 cm mini-column coupled to tandem mass spectrometry. The method was validated for barley, corn, oats, rye and wheat, which represent the majority of grain ingredients in a large number of products destined for human consumption in Ontario, Canada. Detection limits for the various mycotoxins are below current Canadian maximum residue limits. The validated method was tested using internationally recognized proficiency controls and included the analysis of 100 field samples.

P-14 Determination of Pesticide Residues in Vegetables and Fruit Juices using Triple Quadrupole LC-MS/MS

Yang Chen, Fadwa Al-Taher, Rima Juskelis, and Jack Cappozzo; **National Center for Food Safety Technology/IIT**, 6502 South Archer Road, Summit-Argo, IL 60501; (708)563-8172, ychen70@iit.edu

A sensitive QuEChERS (quick, easy, cheap, effective, rugged and safe) method using LC-MS/MS has been developed for analysis of 40 pesticides in vegetables and fruit juice blend samples. Matrix blanks (10 g) were spiked with a mixture of pesticide standards at 10 to 500 µg/kg. The samples were extracted using 1% acetic acid in acetonitrile (10 mL, v/v), followed by addition of ammonium acetate (1 g) and anhydrous magnesium sulfate (4 g). The extract was cleaned up using solid phase extraction with primary secondary amine prior to LC-MS/MS analysis. An Agilent 1200 series LC equipped with a Zorbax extend C_{18} column and a 6460 series Triple Quadrupole MS/MS system was used incorporating thermally focused electrospray ionization. Recoveries of 80-110% were obtained for each pesticide with a relative standard deviation (RSD) of < 5%. Standard curves showed a good linearity (R^2>0.995) across a wide concentration range (10-500 µg/L), with a limit of detection of 0.1 µg/kg and limit of quantitation of 1.0 µg/kg for most pesticides tested. Proficiency tests through the Grocery Manufacturers Association-Food Industry Analytical Chemists Committee showed that the differences between the results obtained in our laboratory and the average results of all participating laboratories were mostly within one standard deviation.

P-15 Flexible, Automated Sample Processing System for Analysis of Drinking Water Using SPE Cartridges and Disks

Thomas K. Dobbs, Jeff Wiseman, and Jennifer Salmons, **J2Scientific**, 1901 Pennsylvania Drive, Suite C, Columbia, MO 65202; (573)214-0472, tdobbs@j2scientific.com

Many of the EPA methods, such as 525.2, approved to monitor water quality in finished or process drinking water involve the extraction of contaminants or specific analytes by use of a prepared C-18 disk or cartridge. The methods require manipulation of large volumes of water, are laborious and time consuming when processing multiple samples and require frequent intervention by the operator. The J2 Scientific PrepLinc™ LVi system advances the automation of these processes through its use of precise large volume sample introduction, bottle rinsing, decreased cartridge drying time and inline sample drying. A full range of disks and cartridges are accommodated. A typical drinking water extraction application for pesticide recovery using EPA 525.2 is presented.

P-16 Ultra-Fast LC/MS/MS Analysis of Acidic Herbicides in Water Samples by Direct Injection: Evaluation of Different MRM Techniques Combined with Polarity Switching

Kristin von Czapiewski,[1] J. Stahl-Zeng,[1] Detlev Schleuder,[1] J. Dahlmann,1 W. Seitz,[2] Henning Blott,[2] Alek Dooley,[3] Rolf Kern; **AB SCIEX**, Small Molecule Applications, 353 Hatch Drive, Foster City, CA 94404; (650)554-7658, rolf.kern@absciex.com
1. Applied Biosystems, Darmstadt, **Germany**
2. Zweckverband Landeswasserversorgung, Langenau, 89129, **Germany**
3. Integrated Gulf Biosystems, Cairo, **Egypt**

Pesticides in general are compounds used to defeat, destroy, repel, or mitigate any pest, unwanted species of plants, fungi, or fungal spores. Pesticide regulations do vary from country to country, thus making global detection rather difficult. Additionally, pesticides used in farming have a tendency to migrate into various water sources. LC/MS/MS trace analysis of drinking, ground, and surface water samples with direct injection becomes an essential tool to investigate in the quality of water. The direct methods presented here offer superior throughput and recovery due to the lack of pre-treatment or other sample preparation techniques.

P-17 Simultaneous Screening Method for Residual Veterinary Drugs in Milk and Honey Using LC-MS/MS after QuEChERs Clean-Up

YunYun Zou, André Schreiber, and Doina Caraiman; **AB SCIEX**, Product Application Laboratory, 71 Four Valley Dr., Concord, Ontario, L4K4V8, **Canada**; (905)660-9005, yunyun.zou@sciex.com

Veterinary drugs are widely used in food animals. The residues left in tissues can lead to variable health problems like antimicrobial resistance and hypersensitivity. Here we present a simple and rapid method to screen for, quantify and identify approximately 90 veterinary drug residues in milk and honey by liquid chromatography coupled to tandem mass spectrometry (LC-MS/MS) after QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) clean-up. Different QuEChERS procedures were evaluated with respect to recovery and matrix effects. It was found that the pH value of honey should be adjusted (addition of sodium hydroxide) before extraction for less matrix effects during ionization. Milk extracts were cleaned up using different dispersive SPE materials. C18 was found to be necessary to efficiently remove fat from the extract resulting in less matrix effects. The approach with C18 only resulted in better recovery compared with mix of PSA and C18. The analysis was performed using a Shimadzu UFLC XR system with a 3 µm particle size C18 column. Compounds were detected using an AB SCIEX 4000 QTRAP® LC/MS/MS system with electrospray ionization in positive and negative polarity. The hybrid triple quadrupole linear ion trap was operated in selective multiple reaction monitoring

mode for quantification, and additionally characteristic MS/MS spectra were collected for compound identification based on mass spectral library searching.

P-18 Pesticide Screening and Quantitation in Cotton Goods

Brian Shofran, April Thomas,[1] Anna Marques,[1] and Andre Schreiber;[2] **AB SCIEX**, 7356 Louise Drive, Fort Myers, FL 33967; (239)482-0514, brian.shofran@absciex.com
1. AB SCIEX, Framingham, MA
2. AB SCIEX, Concord, Ontario, **Canada**

The development of fast and reliable methods for the analysis of pesticide residues is an important field of research. This demand has been stimulated by the continuous public concern over residues in the food supply, in agricultural commodities, and the environment. According to the USDA, in one year alone over 50 million pounds of pesticides were used only in the U.S. cotton fields. Pesticides used on cotton have been linked to ground and surface water contamination, and pollution of drinking water. In this work, an AB SCIEX 5500 QTRAP system was optimized by running a mix of a wide range of pesticides standards. The 16 minute LC-MS/MS method was created using 638 Scheduled MRM (sMRM) transitions, using 2 transitions per analyte, one used for quantitation and the other for confirmation. A variety of plain white cotton T-shirt samples were used in this work. Samples were collected from Brazil, India, China, Canada, and Haiti. The results obtained showed that some samples were positive for some pesticides such as: fenitrothion, diphenilamin, tebufenoxide, oxadixyl, and aldicarb sulfoxide.

P-19a Determination of Aminopyralid in Vegetation by Solid-Phase Extraction and LC-ESI/MS/MS

Jona Verreth, Angela Schaner, and Heidi Hickes; **Montana Department of Agriculture, Analytical Laboratory Bureau**, McCall Hall, MSU, Bozeman, MT 59717; (406)994-3383, jverreth@mt.gov

Aminopyralid was introduced in 2006 as a new member of the pyridine carboxylic family of herbicides. It acts as a growth regulator with selective control of broadleaf weeds in grasses. It is effective at very low rates compared to clopyralid and picloram and is classified by EPA as a Reduced Risk Pesticide due to its lower risk to humans and the environment. To assist regulators with investigations the Montana Department of Agriculture laboratory developed and validated a method to measure aminopyralid at ppb levels in plant tissue. The method adapts the analytical approach of Dow AgroSciences methods that utilize LC-ESI/MS/MS and an isotopically labeled surrogate standard. Two precursor-product ion transitions quantify and confirm results. A 10 g sample is extracted with 0.1 N NaOH and an aliquot is heated to convert the sugar conjugate metabolite to the parent compound prior to clean up on a 30 mg MAX SPE cartridge. Average recoveries for plant tissue ranged from 78-85%.

P-19b Determination of Aminopyralid in Soil by Solid-Phase Extraction and LC-ESI/MS/MS

Jona Verreth, Angela Schaner, Mark Winslow and Heidi Hickes; **Montana Department of Agriculture, Analytical Laboratory Bureau**, McCall Hall, MSU, Bozeman, MT 59717; (406)994-3383, jverreth@mt.gov

Aminopyralid is used on grasslands for selective control of broadleaf weeds. It binds to plant material that is fed to cows/horses and passes through the animals without breaking down. Their manure used as fertilizer can contain trace residues that damage sensitive crops such as beans, tomatoes, potatoes. To assist regulators with investigations the Montana Department of Agriculture laboratory developed and validated a method to measure aminopyralid at ppb levels in soil/manure matrix. The method adapts the analytical approach of Dow AgroSciences methods that utilize LC-ESI/MS/MS and an isotopically labeled surrogate standard. Two precursor-product ion transitions quantify and confirm results. A 10 g sample is extracted with ACN/1N HCL and an aliquot is subjected to clean up on a 30 mg MAX SPE cartridge. Average recoveries for manure ranged from 61-146%.

P-20 Automated QuEChERS Extraction for the Confirmation of Pesticide Residues in Foods using LC/MS/MS

Virgil Settle, Paul Roberts,[2] Peter Stone,[3] Joan Stevens,[4] Jon Wong,[1] Kai Zhang,[1] Fred Foster; **GERSTEL, Inc.**, 701 Digital Drive., Suite J, Linthicum, MD, 21090; (410)218-3913, vasettle@gerstelus.com
1. FDA-CFSAN, College Park, MD
2. Anatune, Ltd., Hardwick, Cambridgeshire, **UK**
3. Agilent Technologies, Santa Clara, CA
4. Agilent Technologies, Little Falls, DE

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 require many manual steps, such as shaking, 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 foods. This poster describes an automated

dispersive SPE cleanup method and LC/MS/MS analysis of QuEChERS extracts prepared with commercially available kits from Agilent. Sample clean-up is achieved using a GERSTEL MPS 2XL Prepstation with an Anatune CF-100 centrifuge. Analytical methodology for confirming the presence of a variety of pesticides in various food samples was developed using an Agilent G6460A Triple Quadrupole Mass Spectrometer. The sensitivity and selectivity of LC/MS/MS allows detection limits of the method to meet acceptance criteria for reporting maximum residue levels (MRLs) as established by regulatory agencies. The ability to automate the 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.

P-21 Optimized Extraction of Paraquat and Diquat from Various Matrices Prior to LC-MS/MS Analysis

Lee Williams,[1] Reese Jones,[1] Helen Lodder,[1] Geoff Davies,[1] Steve Plant,[1] Steve Jordan,[1] Richard Calverley,[1] Claire Desbrow,[1] Gary Dowthwaite,[1] and Russ Katz; **Biotage**, 10430 Harris Oaks Blvd., Suite C, Charlotte, NC; (434)825-6975, Russ.Katz@Biotage.com
1. Biotage GB Limited, Hengoed, **United Kingdom**

Paraquat and Diquat are some of the most widely used herbicides globally. Environmentally, there are concerns due to the toxicity of these non-selective herbicides, not only to non-target plant tissue but also to animal and aquatic organisms and humans. Mixed-mode cation exchange SPE incorporates a hydrophobic polymer and weak cation exchange functionality (WCX). Cleaner extracts are obtained using mixed-mode SPE due to the additional wash step afforded by the dual retention mechanism of the sorbent. Paraquat and diquat have a very strong interaction to the EVOLUTE WCX phase as they have two positively charged quaternary amine functionalities instead of one. We evaluated extraction conditions investigating optimum pH control and elution solvents. As the pH of the SPE conditions increase both paraquat and diquat interaction increases. This results in overall extraction efficiencies decreasing. The elution solvent investigation demonstrates that increasing the polarity of the elution solvent with up to 20% H_2O can increase extraction efficiency. Using 2% formic acid in acetonitrile demonstrates better extraction efficiency compared to 2% formic acid in methanol. Overall the optimum SPE conditions demonstrate extraction efficiency greater than 80% with RSDs less than 10% for both formats.

P-22 Quantitation of Pesticides in Flue-Cured Tobacco by Comprehensive Two-Dimensional Gas Chromatography-Time of Flight Mass Spectrometry (GCxGC-TOFMS)

Joe E. Binkley; **LECO Corporation**, Life Sciences and Chemical Analysis, 3000 Lakeview Ave, St. Joseph, MI 49085; (269)985-5839, joe_binkley@leco.com

Tobacco farmers can increase crop yields with less labor by utilizing pesticides to control insects and weeds. Pesticide residues remaining on the plant after curing can be passed on to the consumer and therefore must be monitored. Tobacco is known to contain thousands of analytes making it an extremely difficult matrix in which to identify and quantitate pesticide residues. Current methodologies incorporate the use of complex, time consuming cleanup techniques to eliminate much of the matrix interference prior to GC-MS analysis. This poster will show the benefits of comprehensive two-dimensional gas chromatography for identification and quantitation of pesticide residues in complex matrices such as tobacco. The increased chromatographic resolution provides the ability to effectively separate pesticides from complex sample matrix while minimizing the need for extensive, time consuming sample cleanup methods. In addition, the cryo-focussing effects of thermal modulation increases detectability allowing lower levels of these pesticide residues to be determined. The use of a Time of Flight Mass Spectrometer provides the ability to acquire full mass range spectra without sacrificing speed or sensitivity. This is beneficial for detecting not only target pesticide residues, but also new and emerging contaminants. Quantitative data from two flue-cured tobacco samples will be shown.

P-23 NOT PRE-DESIGNATED

P-24 Transfer and Speed-up of Methods to Fused-Core™ Particle Columns - EPA Method 1694

Hillel K. Brandes, William H. Campbell, Wayne K. Way, Richard A. Henry, David S. Bell, Carmen T. Santasania, and Ed Mauney; **Supelco/Sigma-Aldric**h, 127 Haselwood Dr, Statesboro, GA 30458; (912)489-8238, ed.mauney@sial.com

EPA method 1694 is used in the determination of pharmaceuticals and personal care products (PPCPs) in environmental samples. These PPCPs have received considerable press attention in recent years, with growing concerns over the presence of these compounds in the world's drinking waters. This method uses solid phase extraction (SPE) and high performance liquid chromatography combined with tandem mass spectrometry (LC-MS-MS) to quantify more than 70 PPCPs in a variety of environmental matrices. In this work, the Group 1 compounds of EPA method 1694 were run on a column containing Fused-Core™ particles. HPLC columns packed with Fused-Core particles or porous particles in the sub-2 µm range can both enhance resolution and speed by producing either higher efficiency (N) for the same column length or equivalent efficiency with a shorter column length. The use of ultra-high performance liquid chromatography (UHPLC) comes at a price of higher pressure when sub-2 µm particles are employed. As an alternative, fused-core silica particles have gained acceptance because of the equivalent performance to particles in the sub-2 µm range. These fused-core columns employ conventional 2 µm frits and operate ruggedly at much lower pressures that are within the operating limits

of conventional HPLC instruments. In this work, Group 1 compounds are shown to exhibit good resolution with MS-MS detection indicating these fused-core columns may be used in this EPA method.

P-25 Method for Analysis of Organophosphorous Pesticides in Produce using Acetonitrile Extraction, Dual-Layer Carbon-Aminopropylsilica Cleanup and GC-NPD Detection

Olga Shimelis, Katherine Stenerson, Michael Ye, Carmen T. Santasania, and Ed Mauney; **Supelco/Sigma-Aldrich**, 127 Haselwood Dr, Statesboro, GA 30458; (912)489-8238, ed.mauney@sial.com

Organophosphorous compounds belong to a large class of pesticides used in agriculture since they are more amenable to environmental degradation in comparison to organochlorine or organonitrogen compounds. Because of their potential health effects, organophosphorous pesticides are of particular concern on produce imported from areas in which they are commonly used. We present here a method for analysis of 65 organophosphorous pesticides from spiked cabbage, green onions, button mushrooms and apples. The extraction was achieved using acetonitrile, with the addition of sodium chloride and magnesium sulfate to induce a salting out effect. A dual layer primary-secondary amine(PSA) / graphitized carbon black (GCB) SPE tube was used for extract cleanup, and the elution solvent contained 35% toluene and 65% acetonitrile for optimum recovery. Solvent exchange into ethyl acetate provided sample extracts with minimal background for GC-nitrogen-phosphorous detection (NPD). Using matrix-matched standards for quantitation, average recoveries for most organophosphorous pesticides spiked at 10 ng/g were in the 60-100% range with average standard deviations of 5% in cabbage and apples, 8% in mushroom, and 12% in green onions.

P-26 Get Connected to The Water Laboratory Alliance!

Marsha Pryor; **Pinellas County Utilities Laboratory**, 1620 Ridge Road Bldg. B, Largo, FL 33778; (727)582-2306, mpryor@co.pinellas.fl.us

What is the Water Laboratory Alliance? In collaboration with partners in the Water Sector, the EPA (Environmental Protection Agency) launched a nationwide laboratory program, the Water Laboratory Alliance (WLA), in September, 2009. This program will increase the preparedness and role of laboratories to respond to water contamination events. The WLA is an integrated nationwide network of laboratories with the analytical capabilities and capacity to support monitoring, surveillance, response, and remediation of intentional and unintentional water contamination events. The WLA structure consists of many national, state and local laboratories, both public and private labs. This extensive network of labs can be easily accessed during a major event such as a terrorist attack or a wide spread epidemic. However, most water contamination events can and should be handled locally. Tools and guidance provided by the WLA can be used locally to investigate possible water contamination as well as for large scale events. For more information on the WLA and member benefits visit http://cfpub.epa.gov/safewater/watersecurity/wla.cfm
Register for the EPA Compendium & WLA at www.epa.gov/erln/join.html
WLA Liaison for the State of Florida: Marsha Pryor (mpryor@co.pinellas.fl.us)

P-27 Determination of Organophosphate and Triazine Pesticides in Water by GC-ECD

Ana Pereira,[1,3] Mónica Teixeira,[1,3] Rui Rangel,[2,3] Cristina Delerue-Matos,[1] and Valentina Fernandes Domingues;[1] [1]**ISEP-REQUIMTE**, Rua Dr. António Bernardino de Almeida 431, Porto, 4200-072, **Portugal**; +351 228340500, vfd@isep.ipp.pt
2. INML, IP – Instituto Nacional de Medicina Legal, **Portugal**
3. CESPU- Cooperativa de Ensino Superior, Politécnico e Universitário, **Portugal**

The analysis of pesticides in water samples is a problem of primary concern for Quality Control Laboratories due to the toxicity level of these compounds and its public health risk. The Council Directive 2078/92/CE regarding the quality of water intended for human consumption states a parametric value of 0.1 µg/L for each individual pesticide and 0.5 µg/L for the sum of all pesticides. The main objective of this work is to develop a sensitive methodology, to quantify organophosphates, triazines and metabolites (atrazine, desethylatrazine, deisopropylatrazine, simazine; terbutilazine, desethylterbuthylazine, malathion, diazinon, dimethoate, chlorpyrifos and azinfos-methyl), using a solid-phase extraction and gas chromatography electron capture detector (GC-ECD). The well water samples have been collected in Esposende, north of Portugal. Esposende, is located in a vulnerable area, with potential for contamination by pesticides. The extraction of all samples has performed with solid phase extraction. Linear calibration curves were obtained to the 11 pesticides. Concentrations ranging from 5 to 500 µg/L were studied. Lichrolut EN/RP-18 100/200 mg and Strata SDB-L 500mg/6mL cartridges were used to concentrate analytes in water samples. The optimization of conditioning, volume breakthrough, recoveries and pH of samples were evaluated.
The authors are grateful to CESPU (Project MSTT) for the material support for this work.

P-28 Determination of Organochlorine Pesticides from Organic and Integrated Pest Management Farming Strawberries using QuEChERS

Vírginia Cruz Fernandes,[1,2] Valentina Fernandes Domingues,[1] Nuno Mateus,[2] and Cristina Delerue-Matos;[1] [1]**CIQ – Centro de Investigação em Química / REQUIMTE**, Rua do Campo Alegre, 687 4169-007, Porto, 4169-007, **Portugal**; +351 220402562, vircru@gmail.com
2. Centro de Investigação em Química, Universidade do Porto, **Portugal**

Organochlorine pesticides (OCPs) are prevalent environmental contaminants with high lipophilicity and long half-lives. They break down slowly and can remain in the environment long after application and in organisms long after exposure. The aim of this study is to develop an analytical method to determine eleven OCPs in strawberries obtained from organic and integrated pest management (IPM) farming. The QuEChERS (EN 15662) procedure involved the extraction of 10 g of homogenized
strawberries samples in a Teflon centrifuging tube with 10 mL acetonitrile. After centrifugation, the supernatant was placed into a clean-up tube containing 150 mg primary secondary amine sorbent plus 150 mg anhydrous magnesium sulfate and 50 mg C18. The supernatant were concentrated and redissolved in n-hexane and finally 2 µL was injected into the GC for analysis. Recoveries obtained were between 54-128% and the limits of detection (3.4-8.7 $\mu g L^{-1}$) of these pesticides were much lower than MRL. The method was applied to analyze four strawberries samples from OF and two from IPM. Finally the proposed method using QuEChERS and GC-MS was successfully utilized for determination of OCPs in strawberries.
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P-29 Determination of Endocrine Disruptors in Portuguese Rivers

Ana Isabel Pereira,[1] Virgínia Cruz Fernandes,[1,2] Catarina Mansilha,[3] Cristina Delerue-Matos,[1] and Valentina Fernandes Domingues;[1] **ISEP-Requimte**, Engenharia Química, Rua Dr. António Bernardino de Almeida 431, Porto, 4200-072, **Portugal**, +351 228340500, vfd@isep.ipp.pt and vircru@gmail.com
2. CIQ Centro de Investigação em Química, Porto, **Portugal**
3. Instituto Nacional de Saúde Dr. Ricardo Jorge, Porto, **Portugal**

Nowadays, the issue of endocrine disruption is, probably, one of the topics in environmental toxicology that receives more attention from the scientific community and regulatory authorities worldwide. Endocrine disrupting compounds (EDCs) comprise a wide variety of pollutants, including some pesticides that occupy a unique position among the chemicals detected in the environment since they are deliberately used. The aim of this study was the development and validation of a multi-residue methodology based on a solid phase microextraction (SPME) coupled with gas chromatography-mass spectrometry (GS-MS) to analyze EDCs pesticides such as: atrazine, dieldrin, aldrin, endrin, iprodione, cypermethrin, deltamethrin, fenvalerate, thiram, HCH, HCB, 2,4-D, alachlor, metoxychlor, endosulfan (I, II) DDD, DDT, DDE, simazine, malathion, vinclozolin, diazinon and lindane. A PDMS fiber was used and some variables (extraction time, desorption time, methanol % and NaCl %) were considered in order to establish optimum conditions. The method was then applied to the analysis of several rivers, allowing the determination of the occurrence and concentration of these compounds in surface water samples from the north of Portugal.

P-30 GPC Clean-Up for Pesticide Residues in Fatty Food

Wendy Rasmussen, David Mazawa, Laszlo Torma, and Uwe Aulwurm;[1] **Pickering Laboratories Inc**, 1280 Space Park Way, Mountain View, CA 94043; (650)694-6700, wendyr@pickeringlabs.com
1. LCTech GmbH, Dorfen, D-84405, **Germany**

Gel permeation chromatography (GPC) is an effective means of sample cleanup prior to analysis. A GPC column and organic solvents are used to selectively pass large macro molecules such as lipids, resins, waxes, and other compounds from sample extracts while retaining the smaller target analyte molecules in the pores of the gel. A large amount of unwanted co-extractives are eliminated. The desired analytes are displaced from the gel with an organic solvent, and depending on the method, are then concentrated to a desired volume on the GPC Ultra. Following clean-up, the solution is analyzed with an appropriate instrument and detector, such as a gas chromatograph with an MSD or ECD. Using the GPC Ultra the laboratory can efficiently and reproducibly clean up a wide range of matrices, from environmental sludge and waste water to foods and fatty tissues in fish or meat. The cleaned extracts can then be analyzed for PAHs, phthalates, azo dyes and more. In this example the system was used to clean a sample prior to analysis of pesticides. We demonstrate excellent results and recoveries for a variety of pesticides in fatty tissues. In our example, this is a sample of rendered chicken fat.

P-31 Student Internships at the US FDA: A Unique Opportunity

Yelena Karaseva, Deb N. Chakravarti,[1] Jamel Sighn,[1] Denise Guajardo,[1] Tenzin Noryang;[1] **FDA ORA Northeast Regional Lab**, 158-15 Liberty Ave, Jamaica, NY 11433; (718)340-7122, ykarasev@ora.fda.gov
1. York College / CUNY,, Department of Chemistry, Jamaica, NY

The Food and Drug Administration (FDA) is a pivotal resource in the health of our nation. Promoting an interest in science and the health enterprise is significant to the missions of both the U.S. FDA and York College – CUNY. Both organizations have a vested interest in improving the quality of and access to scientific research training for minorities, women, and other populations traditionally underrepresented in the scientific and health fields. The York College / U.S. FDA student internship is a great opportunity for instruction and mentorship, while also providing work experience, networking opportunities, academic credit and a stipend. U.S. FDA has an exciting Student Career Experience Program (SCEP) that provides students with the opportunity to gain appropriate experience commensurate to their academic curriculum and career goals. The SCEP program is designed to provide a source of well–trained employees for career entry into the Federal workforce. NRL Pesticide Laboratory has a leadership role in the Internship program. A total of 20 interns had an excellent opportunity to gain all hands on training in a wide variety of methods, tests, and procedures to determine contamination of foods in pesticide analysis.

P-32 Inter-laboratory Validation of a Modified QuEChERS and LC-MS/MS Method for Analysis of >250 Pesticide Residues in Six Fatty Food Products

Yi Lin, Narong Chamkasem,[1] Richard Thompson,[2] Kai Zhang,[3] and Jon Wong;[3] **FDA ORA Pacific Regional Laboratory Southwest**, 19701 Fairchild Ave., Irvine, CA 92612; (949)608-3543, Yi.Lin@fda.hhs.gov
1. FDA ORA Southeast Regional Laboratory, Atlanta, GA
2. FDA ORA Arkansas Regional Laboratory, Jefferson, AR
3. FDA Center for Food Safety and Applied Nutrition, College Park, MD

A slightly modified version of the QuEChERS method was employed in which a phase separation step was incorporated into the method and the grinding time and the centrifugation rate and time were increased. The method was evaluated by analyzing 28 pesticide residues, and includes the optimization of the LC-MS/MS operating parameters towards >250 pesticide residues in fatty food products by multiple FDA laboratories. Avocado, milk, olive oil, fish, shrimp and almond sample composites were extracted by vortex mixing and shaking on a SPEX 2000 Geno grinder. Slightly different LC-MS/MS conditions were optimized at each participating laboratory. The method for determination of 28 pesticide residues in fatty food products was evaluated at three fortification levels (10, 50, and 100 ppb for milk, fish and shrimp; 20, 100, and 200 ppb for avocado, olive oil and almond). The overall recoveries from three fortification levels for six fatty food matrices are satisfactory with most individual recoveries >70%. The demonstrated Limit of Detection (LOD) was 1.0 ng/mL. The demonstrated Limit of Quantification (LOQ) was 10 ppb (or 20 ppb depending on sample matrix) as shown by obtaining acceptable recovery data at those concentration levels. Post-spike extract recoveries averaged >90% which indicates that matrix interferences were adequately minimized. Solvent-based standards can be used during the analysis of the fatty matrices studied. Acquisition of the validation data towards the analysis over 250 pesticide residues is in progress.

P-33 Rapid Dispersive SPE Multiresidue Method for Analysis of Polar and Nonpolar Pesticides using Acetone

Olusegun J Ajayi, Irene Cassias, and Eugene Chang; **FDA ORA Pacific Regional Laboratory Southwest**, 19701 Fairchild, Irvine, CA, 92612; (949)608-2981, olu.ajayi@fda.hhs.gov

The need for a rapid, simple, eco-friendly and efficient extraction process with minimal cost of operation is necessitated in ever changing dynamic environment. This new procedure is relatively low in labor capital and energy efficient compared to other extraction methods previously employed in FDA. Los Angeles Micro Method (LAMM) method does sample extraction process by extracting pesticide residues from 10 g of finely chopped sample by vortex mixing and centrifuging with 10 ml of acetone. Water removal and extract cleanup is performed by salting out with fructose, magnesium sulfate, sodium chloride, PSA and C-18 mixture through dispersive SPE process. The method shows similar low limit detection results when compared with Quick, Easy, Cheap, Effective, Rugged and Safe (QuEChERS) and Luke methods. The proposed method produces high recovery results with a clean and clear extract that minimizes frequent GC/MSD maintenance schedule.

P-34 Monitoring for Nontargeted Veterinary Residues in Food Samples using Quadrupole Time-of-Flight (Q-TOF) LC/MS

Sherri B. Turnipseed, Joseph M. Storey,[1] and Susan B. Clark;[1] **FDA Animal Drugs Research Center**, PO Box 25087, Denver Federal Center, Denver, CO 80225; (303)236-3072, sherri.turnipseed@fda.hhs.gov
1. U.S. Food and Drug Administration, Denver Laboratory, Denver, CO

Our laboratory is currently utilizing a quadrupole time-of-flight (Q-TOF) LC/MS for the analysis of nontargeted veterinary drug residues in milk and aquacultured samples. A Q-TOF mass spectrometer has the advantage of being able to obtain full scan data on potentially

an unlimited number of compounds. For confirmation, additional structural information can be obtained by collecting MS/MS data. Fluoroquinolones (FQs) antibiotics are not allowed for use in food producing animals in the U.S. because of concerns regarding antibacterial resistance. Often, residues of FQs are monitored initially by LC-fluorescence followed by confirmation by LC-MS. We utilized a Q-TOF LC-MS method for aquacultured samples to not only confirm target analytes, but also to monitor for other compounds. For example, trimethoprim was detected and identified in a sample of frog legs along with several FQ residues. The Q-TOF instrument has also been used to analyze residues in incurred milk samples. In addition to ciprofloxacin, a second metabolite was found in milk from cows dosed with enrofloxacin. This metabolite matched the accurate mass and had a similar retention time to pefloxacin, another FQ, but the MS/MS spectra indicated that the metabolite was a different compound.

P-35 Tubes or Tips?

Lynda Podhorniak; **EPA Analytical Chemistry Branch**, 701 Mapes Road, Fort Meade, MD 20755; (410)305-2926, podhorniak.lynda@epa.gov

The introduction of the QuEChERS method provided undeniable progress in sample preparation methods for pesticide residue analysis. The increased speed in sample preparation was welcomed by many surveillance laboratories which analyze thousands of samples each year. Using QuEChERS, it was possible for two chemists in our lab to analyze 4,000 fruit samples over a period of only 2.25 years for the N-methyl carbamate, FHCl. This rate of sample analysis would not have been possible using traditional multiresidue methods. In a quest for even more speed and greater sample throughput, another technology has become available which uses a tip instead of a QuEChERS tube for sample cleanup. This approach appears to have potential to further streamline sample preparation. If $MgSO_4$, PSA and GCB in a 15 mL tube provide a sufficient sample cleanup for many matrices and many analytes, then shouldn't $MgSO_4$, PSA and GCB in a tip also provide a sufficient cleanup? The EPA Analytical Chemistry Lab evaluated the effectiveness of the DPX tip approach and compared the DPX tip cleanup to the QuEChERS dispersive tube cleanup for the sample preparation of three different fruit matrices analyzed for FHCl.

P-36 Disposable Pipette Extraction (DPX) Tips for Rapid Cleanup for Simultaneous Analysis of Melamine and Cyanuric Acid using GC/MS/MS and LC/MS/MS

William E. Brewer, Pakritsadang Kaewsuya, Stephen L. Morgan, Alexander J. Krynitsky,[1] and John A.G. Roach;[1] **University of South Carolina**, Dept. of Chemistry and Biochemistry, 631 Sumter St., Columbia, SC 29208; (803)622-9570, brewer@chem.sc.edu
1. FDA Center for Food Safety and Applied Nutrition, College Park, MD

Numerous methods of analysis for melamine and cyanuric acid have been developed. Most methods require two separate extraction and cleanup schemes to analyze both compounds. Some methods have been developed that require essentially no cleanup, but these methods have limitations due to dilution and matrix effects. We present a novel method for rapid cleanup using disposable pipette extraction (DPX) tips. Loosely contained sorbent is placed inside a pipette tip and contained with a screen and upper barrier. This device allows for a convenient dispersive solid-phase extraction procedure that simply involves 3 steps: 1) aspirate sample solution into DPX tip; 2) aspirate air into DPX tip to cause mixing of the solution with the sorbent via air bubbles; 3) dispense "cleaned up" extract for subsequent analysis. We have analyzed extracts of milk products using this cleanup method with GC/MS, GC/MS/MS and LC/MS/MS. Although LC/MS methods do not require chemical derivatization, they do require the use of HILIC columns which present other potential problems. Detection limits less than 20 ppb in whole milk have been achieved using GC/MS/MS. Analysis of other sample matrices are presented, including infant formula and chocolate milk. Complete automation methods for GC/MS and LC/MS are also presented.

P-37 Determination of Nitrofuran Metabolites in Shrimp by LC/MS/MS

Lin Ye, Narong Chamkasem, Anthony Williams, Jean-Marie D. Dimandja;[1] **FDA ORA Southeast Regional Laboratory**, 60 8th St. NE, Atlanta, GA 30309; (404)253-1200 ext. 5449, lin.ye@fda.hhs.gov
1. Spelman College, Atlanta, GA

The method presented here is a modification of the method developed by the U.S. Food and Drug Administration (FDA) Center for Veterinary Medicine (CVM) previously validated for determining nitrofuran metabolites in shrimp [(2005) J. Agric. Food Chem. **53**, 8934-8939]. The modified method uses liquid chromatography/tandem mass spectrometry (LC/MS/MS) with a positive electro-spray interface. The main attributes of this method are a higher throughput sample preparation protocol, and improved selectivity and sensitivity. The limit of detection (LOD) and limit of quantification (LOQ) are 0.01–0.15 ng/g and 0.01–0.30 ng/g. Average recoveries of the nitrofuran metabolites at 3 different levels are 62%, 91%, 90% and 84% for semicarbazide (SCA), 3-amino-2-oxazolidinone (AOZ), 1-ami-nohydantoin (AHD) and 3-amino-5-morpholinomethyl-2-oxazolidinone (AMOZ), respectively. The precision (RSD) of 6 replicates at 1 ppb level are 6.35%, 4.25%, 1.53% and 2.35% for SCA, AOZ, AHD and AMOZ, respectively.

P-38 Development of a Multi-Residue Method for the Determination of Steroid Hormones in Bovine Muscle using LC-MS/MS

Tricia L Johnson; **Food & Drug Administration, Office of Research**, 8401 Muirkirk Road, Laurel, MD 20708; (301)210-4651, tricia.johnson@fda.hhs.gov

Steroid hormones are used in the food industry as growth promoters to improve feed efficiency and weight gain in animals. Tracking the use and abuse of steroid hormones have garnered wide attention in the US and abroad. Illegal use of steroid hormones in food-producing animals could pose safety concerns for human consumption. For this reason, we are developing a multi-residue method for determining the steroid residues in bovine muscle at 1 ng/g using LC-MS/MS. In this procedure, muscle tissue is fortified with a mixture of 16 steroid hormones, including testosterone, diethylstilbestrol, and progesterone, followed by sample cleanup on strong anion exchange solid phase extraction (SPE), on C_{18} SPE, and by liquid-liquid partitioning. The purified extracts are analyzed on LC-MS/MS with polarity switching for simultaneous detection of positive and negative ions. Data of 6 replicates of fortified samples are evaluated for method accuracy and precision, and results will be reported.

P-39 NOT PRE-DESIGNATED

P-40 Evaluation of Several Columns and Solvents for Post-Extraction GPC Clean-Up of Fish Tissue Prior to PBDE or PCB Analysis

Michael Halvorson, Thomas Gibson,[1] Carol Buelow,[1] and Curtis Hedman;[1] **Gilson, Inc.**, 3000 Parmenter St., P.O. Box 620027, Middleton, WI 53562; (608)836-1551, mhalvorson@gilson.com
1. Wisconsin State Lab of Hygiene, Madison, Wisconsin

Gel permeation chromatography (GPC) is an effective and efficient post-extraction clean-up method for the removal of high molecular weight interferences such as lipids, proteins, pigments, natural resins and cellular components from sample extracts prior to analysis by GC, GC/MS, HPLC or LC/MS. GPC clean-up is often used for the clean-up of fish tissue extracts with high lipid content prior to analysis for halogenated compounds such as PBDEs, PCBs and chlorinated pesticides. Traditionally, post-extraction GPC clean-up of fish tissue is accomplished using a low pressure glass column containing Envirobeads® SX-3 resin and a mobile phase of either methylene chloride or 1:1 methylene chloride:cyclohexane. While this column is highly efficient in the removal of lipids, it has the disadvantage of relatively slow throughput per sample extract. The purpose of this study was to evaluate alternative columns and solvents for the post-extraction clean-up of fish tissue extracts that have faster throughput compared to the traditional GPC clean-up column prior to analysis for PCBs or PBDEs. Comparison of GPC calibrant elution times, lipid loading capacity and PCB/PBDE recoveries from fish tissue for each column and mobile phase were determined.

P-41 The MaxSignal® Trifluralin ELISA Kit is a Competitive Enzyme Immunoassay for the Quantitative Analysis of Trifluralin in Fish and Meat

Lance Ford, Zhenglai Fang, Jessica Zabloski, and Rick Valdez; **Bioo Scientific Corp.** ,3913 Todd Lane Suite 312, Austin, TX 78744; (512)707-8993, lford@biooscientific.com

Trifluralin is a selective, pre-emergence dinitroaniline herbicide used to control many annual grasses and broadleaf weeds in a large variety of fruit, nut, vegetable, and grain crops. Trifluralin can persist in soils for many months after it is used and can leach into water where it is highly toxic to aquatic animals (fish and invertebrates). Antibody-based ELISA methods are a sensitive, high-throughput alternative to GC-MS and HPLC based detection techniques for herbicide residues. We will present data illustrating that the MaxSignal® Trifluralin ELISA Kit, a competitive enzyme immunoassay, offers sensitive, quantitative analysis of trifluralin in fish and meat. This technique is inexpensive and highly specific, which makes it preferable to other methods for herbicide residue screening applications. We will also describe the work we performed to streamline the sample preparation techniques to enable a compatible format for ELISA.

P-42 Determination of Selected Persistent Organic Polluting Pesticides in Water using Automated SPE and GC/ECD

Robert S. Johnson, and Bob Riggs; **Horizon Technology**, 45 Northwestern Dr., Salem, NH 03079; (603)893-3663, rsjohnson@horizontechinc.com

Pesticide pollution is a subject of global concern and although many countries have now banned the use of persistent organic pesticides, they linger in the environment and can contaminate water sources. The analysis of trace levels of pesticides in water has proved challenging. Traditional methods of extraction are labor intensive, use large amounts of chlorinated solvents, have matrix interfering emulsions, require contaminant free glassware, and a solvent exchange to hexane. The EU's requirement to eliminate chlorinated solvents adds to the challenge. The pesticides analyzed in this study were part of a mix consisting of DDT, DDD, DDE, aldrin, dieldrin, endrin, endrin aldehyde, endrin ketone and heptachlor in the low µg/L (ppb) range. A new extraction method using hexane as the extraction solvent has yielded results with less solvent use, elimination of the solvent exchange step, reduced glassware use, faster

extractions with highly particulate samples, and more consistent and reproducible results, while also conforming to the EU standards. The method and data will be presented.

P-43 Optimization of Solid-Phase Extraction Combined with Centrifugation for the Determination of Polycyclic Aromatic Hydrocarbons in Water

Abir Kouzayha, Mouhammad AL Iskandarani, Bilal Nsouli, Helene Budzinski[2], and Farouk Jaber;[1] **Lebanese Atomic Energy Commission**, National Analysis of Pesticides and Organic Pollutants Laboratory, PO Box 11-8281 Riad El Solh 1107 2260 Beirut, **Lebanon**; 00961-1-450811, riba0@hotmail.com
2. Université Bordeaux 1, CNRS, ISM, Laboratory of Physico- and Toxico-Chemistry, Bordeaux, **France**

A fast and reliable method for the determination of polycyclic aromatic hydrocarbons (PAHs) in water samples by solid-phase extraction (SPE) combined with centrifugation has been developed and optimized. A silica based C18 cartridge was used, parameters affecting the extraction procedure such as type and volume of the elution solvent, breakthrough volume of percolated water sample, drying of sorbent and concentration of eluate have been studied. The matrix effects were evaluated as well through the salt addition and the analysis of spiked superficial and seawater. The novelty of this work was the investigation of the use of centrifugation technique in both drying and elution steps. When combined with centrifugation, the time for sorbent drying was reduced to several minutes, and the volume of the elution solvent was also minimized compared to the ordinary SPE procedure. The small volume of eluate obtained permitted a rapid solvent concentration using nitrogen gas and reduced majorly the losses of the more volatiles compounds. With this developed procedure, analytical recoveries and linearity for aqueous matrices were satisfactory. Finally, the method has been applied to the determination of PAHs in surface seawater samples collected along the Lebanese coast in the eastern part of the Mediterranean basin.

P-44 Effect of Seed Weight on Pesticide Residues Estimation in Stone Fruits

Kazuaki Iijima; **The Institute of Environmental Toxicology, Chemistry Division**, 4321 Uchimoriya-machi, Joso-shi, Ibaraki, 303-0043, **Japan**; 81-297-27-4516, iijima@iet.or.jp

The purpose of this study is to provide the relation of residue levels determined by the Japanese and international definitions of analysis sample of stone fruits. Sample weights of fresh and seeds of stone fruits such as peach, ume (Japanese apricot), cherry, sumomo (Japanese plum) and nectarine were investigated from the previous field trial data. The correction coefficients were deduced, which can be used for calculation of the residues levels expressed on the whole commodities from the analytical values of them without seeds. The calculated correction coefficients ranged from 0.88 for ume to 0.96 for sumomo. The results indicate that calculated residue levels of whole commodities (according to the international standard) are slightly lower than the actual concentrations without seeds (according to the current Japanese regulation). Small commodities such as cherry and ume are more influenced by the calculation procedure to convert whole commodity basis.

P-45 Large Volume Injection for Gas Chromatography using a Commercially-Available Unmodified Splitless Injector

Jack Cochran, **Restek Corporation**, 110 Benner Circle, Bellefonte, PA 16823; (814)353-1300, jack.cochran@restek.com

Magni and Porzano described Concurrent Solvent Recondensation Large Sample Volume (CSR-LV) Splitless Injection with a special low dead-volume injector, modified septum head to reduce septum temperature, and the ability to close the septum purge duringinjection. They achieved excellent repeatable, linear results for injections up to 35µL. Principles of CSR-LV include fast injection with liquid band formation into a liner containing glass wool, a pre-column press-fitted to the analytical column, and starting oven temperature below the boiling point of the solvent. In this work, for the first time to my knowledge, a standard Agilent GC split/splitless injector was employed with a low-cost, commercially-available, single-taper liner with wool and a 0.53mm pre-column press-fitted to a typical GC column to achieve CSR-LV. No special septum head cooling was necessary and the septum purge remained open during injection and GC. Repeatable and linear results were achieved for hydrocarbons and PAHs with injection volumes up to 25µL. CSR-LV was also used with GCxGC-TOFMS to increase detection limits for pesticides in dietary supplement extracts. Experimental details will be given to allow users to employ CSR-LV with conventional Agilent split/splitless injectors.

P-46 Large Volume Injection on Split/Splitless Inlets "The Pauper's Upgrade"

Lee Miller, Raymond Allum, and Jack Cochran;[1] **Florida Dept. of Agriculture and Consumer Services**, 3125 Conner Blvd., Tallahassee, FL 32399; (850)617-7500, millerl@doacs.state.fl.us
1. Restek Corporation, Bellefonte, PA

In today's laboratory we are being asked to do more and more. The only way to meet the demands is to become more efficient. Utilizing existing resources in the lab can achieve this goal. Increasing injection volumes from 1 µL all the way up to 25 µL ("fast" injection

speed) can increase sensitivity on GC/MS TOF. Typically this is accomplished by purchasing a large volume injection inlet or PTV. Utilizing a split/splitless inlet technique coupled with GC TOFMS, a linear response was produced for a large variety of temperamental compounds, across an entire range of injection volumes, up to and including 25 µL.

P-47 QuEChERS Approach with GCxGC-ECD and GC-TOFMS for the Analysis of Pesticides in Dried Ginseng

Michelle Misselwitz, Jack Cochran, Julie Kowalski, Rick Lake, and Jason Thomas; **Restek Corporation**, 110 Benner Circle, Bellefonte, PA 16823; (814)353-1300, michelle.misselwitz@restek.com

In 2003 and 2004, the US FDA sampled and analyzed imported and domestic ginseng, which is used as a dietary supplement, for pesticide residues. Many of the ginsengs contained pesticides in excess of the EPA Tolerance/ FDA Enforcement Levels, mostly organochlorine pesticides such as hexachlorocyclohexanes, chlorinated benzenes and their derivatives, and even DDT and its analogues. The analytical guidance for sample preparation was to use the Pesticide Analytical Manual (PAM) procedures for organochlorine and organophosphorus pesticides or other appropriate methodology. Unfortunately, the PAM procedures are solvent, glassware, and labor intensive. We obtained samples of dried ginseng labeled "Chinese White" and "Korean Red" and used the QuEChERS (Quick – Easy – Cheap – Effective – Rugged – Safe) approach to prepare the samples for GC-TOFMS and GCxGC-ECD analyses, comparing the results against those previously generated with a different extraction procedure and GC-MS with selected ion recording and GC-MS/MS. QuEChERS allowed a rapid sample preparation and both GC-TOFMS and GCxGC-ECD results compared favorably with those from the highly selective GC-MS/MS.

P-48 Analysis of Polycyclic Aromatic Hydrocarbons in Infant Formula using the QuEChERS Approach Combined with Both Liquid and Gas Chromatography

Julie Kowalski, Michelle Misselwitz, and Jack Cochran; **Restek Corporation**, 110 Benner Circle, Bellefonte, PA 16823; (800)356-1688 ext 2354, julie.kowalski@restek.com

Polycyclic aromatic hydrocarbons (PAHs) are a group of chemicals that are known or suspected carcinogens. Exposures to PAHs usually occurs by eating charbroiled foods, inhaling burning coal, tar, garbage, exhaust fumes and smoke but are also present in medicines, plastics and pesticides. HPLC, UHPLC and GC stationary phases and formats were evaluated and methods optimized for selectivity and decreased analysis time while maintaining column efficiency. The target analyte list evaluated includes the 18 EPA 610/8100 mandated target PAHs along with the 15 EU 256/2005 recommended PAH analytes. The QuEChERS approach was evaluated as a sample preparation method to help achieve low level detection of PAHs. The fast UHPLC-UV analysis proved ideal for a screening method. GCMS analysis provided mass spectral conformation in addition to retention time matching. Single ion monitoring (SIM) also aided in low level detection. Infant formula samples were tested and PAHs were detected below the EU limit of 1 µg/kg.

P-49a Evaluation of Various Approaches for the Estimation of Measurement Uncertainty in Multi-Residue Methods for Fruit and Vegetables

A. Valverde, A. Aguilera, P. Medina-Pastor,[1] M. Gamon,[1] T. Pihlström,[2] S. Masselter,[3] and A.R. Fernández-Alba[1]; Pesticide Residues Research Group, **University of Almeria**, 04120 Almeria, **Spain**; amadeo@ual.es
1. EURL-FV - European Union Reference Laboratory for Pesticide Residues in Fruit and Vegetables
2. National Food Administration, Uppsala, **Sweden**
3. AGES Competence Centre for Residues of Plant Protection Products, Innsbruck, **Austria**

Practical (top-down) approaches appears to be the most suitable for evaluation of measurement uncertainty in pesticide residue testing laboratories, where the analytical procedures are routinely applied to a large number of pesticide/food combinations (multi-residue methods), and these methods include a multitude of steps and factors difficult to define and to integrate into the down-top evaluation of measurement uncertainty. There are different ways of estimating uncertainty based on the top-down approaches: within-laboratory reproducibility, interlaboratory validation data, using Horwitz equation, using FFP-RSD estimation of 25%, or using results from proficiency tests. Upon these, there are also guidelines from different bodies commenting the different ways of calculating uncertainty. The Codex Alimentarius Commission has established some guidelines on estimation of uncertainty of results (CAC/GL 59-2006), and the Codex Committee on Pesticide Residues has proposed to further develop a specific guidance for the application of empirical measurement uncertainty concepts in the field of pesticide residue analysis (ALINORM 09/32/24, Appendix X) [1]. We present in this work are specific examples of uncertainty estimations based on precision and bias data obtained from within-laboratory method validation studies, quality control, or proficiency tests of laboratories analysing multi-pesticide residues in fruits and vegetables, and following the ALINORM 09/32/24 (Appendix X) draft proposals.

[1] Codex Alimentarius Commission ALINORM 09/32/24 (Appendix X): Proposed draft revision of the guidelines on the estimation of uncertainty results for the determination of pesticide residues (CAC/GL 59-2006) at step 3

P-49b Pesticide Residue Analyses of Fruit Juices by Direct Injection in LC-MS/MS Minimizing Matrix Effects

Carmen Ferrer, M.J. Martínez-Bueno, Ana Lozano, Tiziana Pacchiarotta, and Amadeo Fernandez-Alba; **Pesticide Residue Research Group. European Reference Laboratory EURL. University of Almeria**. 04120 **Spain**; amadeo@ual.es

Fruit juices are consumed daily in the European Union countries, and monitoring of pesticide residue levels in juices is essential, especially taking into account its high consumption by children. However, matrix effects can severely compromise quantitative analysis of the compounds at trace levels as well as method reproducibility. A possible approach to avoid the effects can be dilution of the extracts for analysis using highly-sensitive analytical systems in the market. For this work, three different fruit juices (orange, pineapple and peach juice) were selected to develop an analytical method for the analysis of 50 pesticides by direct injection in LC-MS/MS. The preparation of the samples was very simple: an aliquot of the juice was centrifuged and filtered, and then it was diluted 10-fold prior to analysis. Validation of the method was carried out in accordance with EU guidelines [1]. Calibration curves covering two orders of magnitude were performed, and they were linear over the concentration range studied for all the matrices (from 0.1 to 100 µg/kg). Practical detection limits (LOD) were in the low µg/kg range, far below the maximum residue levels (MRLs) of the EU regulations, which don't set specific MRLs for juices. Repeatability of the instrumental method was studied in all matrices, obtaining good intra- and inter-day RSDs. The proposed method was applied to 55 real fruit juice samples purchased in different local markets in order to validate the suitability for routine analysis. 56% of the analysed samples gave positive results (higher than the practical LOD).
[1] Document SANCO/10684/2009 Method validation and quality control procedures for pesticide residue analysis in food and feed.

P-50 Update on Proposed and Approved ASTM Methods for Liquid Chromatography/Mass Spectrometry of Interest to US EPA and the Revival of the LC/MS Library Project

Lawrence B. Zintek; **US EPA Chicago Regional Lab**, 536 South Clark Street, Chicago, IL 60605; (312)886-2925, zintek.lawrence@epa.gov

Update on new standardized methods for qualitative and quantitative determination for chemicals of national significance. This presentation will demonstrate why and how we are developing quick and robust liquid chromatography tandem mass spectrometry (LC/MS/MS) methods for analysis of pharmaceuticals, pesticides, and degradation products of chemical warfare agents. These methods focus specifically on analyzing organophosphonic acids, carbamates, rodenticides, pesticides, ethanolamines, thiodiglycols, and bisphenol A in water samples and revival of the LC/MS library work that was presented previously at FPRW. The process for approval of test methods into ASTM International will be discussed.

P-51 Grain and Grain Dust Matrix Matching: Benefits and Pitfalls

Robert D. Trengove, Bruce Peebles, Katherine Roussety, and Sze Bong; **Murdoch University, Separation Science & Metabolomics Laboratory**, South Street, Perth, WA, 6150, **Australia**; +61893607639, r.trengove@murdoch.edu.au

Analyte degradation with gas chromatography based analysis and matrix suppression in electrospray with liquid chromatography based analysis represent two key challenges in residue analysis and the drive to improve limits of detection and therefore improve the quality of food to table. For GC/MS analysis, the use of matrix matched standards and the use of analyte protectants represent two general approaches that have had significant success, and generally negate the need for a standard addition approach. In particular, matrix matched standards allow GC/MS analysis of some compounds which are generally regarded as "non-GC compatible," providing further confidence in identification and quantification of positives when these compounds are screened using both a GC and LC based analysis. The use of stable isotope dilution represents a very costly and unsustainable alternative for large scale multi-residue analysis of food. Results for the GC/MS analysis of grains illustrating the success of matrix matched standards for single grain analysis will be presented. Grain dust analysis will be used to demonstrate the necessity for matrix matched internal standard sets rather than a single internal standard and the critical role for specific matrix matched standards. The minimization of false positives using matrix specific transitions will also be illustrated.

P-52 Optimization of HPLC/API-MS/MS Analytical Conditions in order to Reduce Matrix Interferences in the Simultaneous Detection of more than 30 Mycotoxins in Cereal Grains

Robert D. Voyksner and Jennifer Sealey-Voyksner, **LCMS Limited**, PO Box 27228, Raleigh, NC 27611; robert_voyksner@lcmslimited.com

Mycotoxin contamination of grain and food products is becoming an increasingly important issue in food safety. LC/MS techniques have been demonstrated to meet the challenging task of detecting these toxins at ppb levels in grain and foods, but matrix effects often limit the effectiveness of the technique. This is particularly evident when using simple acetonitrile or methanol extractions to measure a large number of mycotoxin residues simultaneously in a sample. This presentation will evaluate matrix effects to determine the appropriate analytical conditions, including: 1) The mode of ionization (ESI, APCI or APPI) and detection (positive or negative) that offers the best signal to noise and the least matrix effects; 2) The most intense parent to product ion transitions which allow optimal sensitivity and specificity; and 3) The relationship between chromatographic analysis time and matrix effects. Results demonstrate

that both positive and negative ion detection are required in order to detect all target mycotoxin residues. Most mycotoxins produced at least 3 intense MS/MS product ions, however, several residues produced only the $[M+OAc]^-$ ion (m/z 59), which was found to be prone to matrix interference. ESI was generally found to be the most effective and sensitive mode of ionization, while APCI appeared to show the least overall matrix effects. Matrix suppression from grain extracts was the most predominant with short (3 min) gradient elution times, using ESI positive ion detection conditions, thus resulting in a lower overall residue recovery (<66%). Longer analysis times (12 and 28 min) resulted in better overall recoveries (86% and 92% respectively).

P-53 State of Texas Approval for Organochlorine and Organophosphorus Pesticides by USEPA Method 8270 Full Scan LVI-GCMS

Larry D. Garretson, Dehrsing Luu[1], John Dupont[1], and Fred Feyerherm[2]; **Scientific Instruments**, 2200 Spanish Oak Trail, Round Rock, TX 78681; (512)259-8298, lgarretson@aol.com
1. DHL Analytical, Inc., Round Rock, TX
2. Agilent Technologies, Houston, TX

Laboratories have been analyzing pesticides by USEPA Methods for many years. The methods require ECD dual column confirmation of pesticides. Analysts are challenged to determine if peaks meet criteria as confirmed pesticides. While the ECD is very specific, many matrix components cause significant interferences and complicate data interpretation. Using GCMS to provide full scan spectra of potential pesticide containing samples is extremely desirable. Past GCMS sensitivity levels were insufficient to meet ECD method detection limits. Presented data includes the analytes Chlordane/Toxaphene. Improved MS sensitivity and programmable injector technology, allows the GCMS in full scan acquisition mode to achieve ECD detection levels. This paper focuses on the technical approach implementing ECD methodology to GCMS-based Method 8270. Configuration includes Agilent 5975C, Trace Ion Detection Mode, High Capacity Turbo-molecular Pump, Triple Axis Detector and ATAS GL International Programmable Temperature Vaporizer Injector. Recent data will also be shown utilizing the Agilent Multi Mode Inlet. The process of submitting data for 'Field of Accreditation" approval by the Texas Commission on Environmental Quality via Proficiency Testing Providers under NELAC supervision will be presented.

P-54 Application of Chemical Ionization GC/MS/MS for Trace Analysis of Pesticides in Foods

Philip L. Wylie and Melissa Chruley;[1] **Agilent Technologies**, 2850 Centerville Rd., Wilmington, DE 19808-1610; (302)633-8416, phil_wylie@agilent.com
1. Agilent Technologies, Department of Marketing, Santa Clara, CA

The detection limits for most pesticides analyzed by electron impact triple quadrupole GC/MS/MS are well below 10 pg on column (10 ppb for a 1-µL injection). In general, selectivity is improved when analyte precursor ions are abundant and have the highest possible *m/z*. However, some pesticides lack an abundant precursor ion at high mass so the selectivity is reduced and matrix interferences become more apparent. Chemical ionization normally results in much less fragmentation and the molecular ion (or pseudo molecular ion) is often the base peak. The objective of this work is to apply negative chemical ionization (NCI) on a GC/QQQ instrument to the GC/MS/MS analysis of pesticides with the goal of improving method sensitivity. NCI is an extremely sensitive technique for compounds that have electronegative functional groups, and many pesticides are in this category. Our assumption was that using NCI would offer the possibility of using the molecular ion or another large fragment as the precursor for MS/MS. The NCI GC/MS/MS method was applied to a set of pyrethroid pesticides containing halogen functional groups. Samples of spinach and apple, extracted using the QuEChERS method, were analyzed by NCI GC/MS/MS using both methane and ammonia reagent gases. Ammonia gave ca. 10 fold greater sensitivity for these compounds in the MRM mode. Using this method pyrethroids could be quantified in the ppt range.

P-55 Rapid Multi-Residue Analysis of Pesticides in Honey using a Buffered Acetonitrile Extraction and Dispersive Clean-Up

Jonathan V Pelt, Amy Brown, and Jason Stepp; **Dept. of Agriculture and Consumer Services, Chemical Residue Lab**, 3125 Conner Blvd. Lab #3, Tallahassee, FL 32399, (850)617-7500, peltj@doacs.state.fl.us

A rapid and inexpensive multiresidue method for pesticides in honey is presented. Extraction of a 15 g sample, with the addition of 15 mL of deionized water and 15 mL acetonitrile was followed by buffering with magnesium sulfate, sodium chloride, sodium citrate dehydrate and disodium citrate. Acidification with formic acid prior to dispersive clean up with aminopropyl and magnesium sulfate was employed to stabilize base sensitive pesticides such as chlorothalonil. Extracts were concentrated to 2 g/mL. Analyses were conducted by GC/MS, GC/XSD and LC/QqQ/MS. Method accuracy, repeatability and linearity were performed for 180 compounds in honey. Typical recoveries were between 70-130% and CVs were <20%. This resulted in 159 compounds being validated. This was a significant increase in compounds compared to the prior method that only screened for 10 compounds. Supply cost per sample was reduced to 46% of the previous method, and time to extract a batch of 24 samples was reduced by 75% compared to the prior method which used a 20 g sample, 125 mL of acetonitrile, multiple SPE columns and additional instrumentation.

P-56 Comparison of Matrix Effects on QuEChERS and Dual-Layer SPE Method of 70 Pesticides from Six Agricultural Products using Triple-Quadrupole Mass Spectrometer

Yu-Ri Lee, Guk-Tak Han, and Kyu-Seung Lee;[1] **National Agriculture Products Quality Management Service (NAQS), Chung-Nam Province**, 495 Bomunno, Jung-gu, Daejeon, Chung-Nam, 301-825, **Republic of Korea**; +82-42-226-6080, lily0517@naqs.go.kr
1. Chung-Nam National University, Daejeon, **Republic of Korea**

Two sample preparation methods, QuEChERS and dual-layer SPE, were compared with matrix effect using a triple-quadrupole mass spectromer. Seventy pesticides were fortified at 0.05 and 0.5 mg/kg as low and high level concentrations. QuEChERS preparative samples were colored by the pigment in each matrix, whereas most pigments were removed by dual-layer method. Because of the matrix effect, 25 and 10 pesticides were not detected in low level concentration using QuEChERS and dual-layer, respectively. Sensitivity of dual-layer method was observed 10-20 times higher than QuEChERS. The order of difference on sensitivity between methods was appeared apple, grape, lettuce, carrot, pimento and sesame, in turn. Sesame having plenty of oil was detected on similar sensitivity in both methods because of insufficiently matrix removed. Difference of sensitivity between two methods was divided in 3 parts according to chemical property for compounds. Pesticides of log P between 1.5 to 4.2 treated dual-layer were more effective for co-eluent removal rate than polar and non-polar pesticides.

P-57 Characteristics of Pesticide Residues and Dissipation in Mushrooms During Growth

Guk-Tak Han, Yu-Ri Lee, and Kyu-Seung Lee;[1] **NAQS, MIFAFF, Chung-Nam Province**, 495 Bomunno, Jung-gu, Daejeon, Chung-Nam, 301-825, **Republic of Korea**; +82-42-226-6080, hankook@naqs.go.kr
1. Chung-Nam National University, Daejeon, **Republic of Korea**

Residue and dissipation pattern of pesticide were investigated and biological half-lives were established in mushrooms and oyster mushrooms. Diflubenzuron, teflubenzuron used in oyster mushrooms and diflubenzuron, prochloraz used in mushrooms were selected and applied on the compost. Residues were quantified after sampling in twice a day. Biological half-lives of diflubenzuron and teflubenzuron were 1.32, 2.12 days at the recommended dose and 2.06, 1.72 days at the double dose in oyster mushrooms. Whereas 1.58, 2.39 days at recommended dose and 2.83, 4.14 days at the double dose were showed for diflubenzuron and prochloraz in mushrooms, respectively. Diflubenzuron was calculated shorter half-life than other pesticides in both types of mushroom. The pesticide residue was affected by amount of uptake from compost and improvement of weight in mushrooms especially.

P-58a Identification of Pesticide Residues in Essential Oils by QuEChERS Extraction and GC/MS Analysis

Joan M. Stevens; **Agilent Technologies**, Department of Consumables and Supplies, 2850 Centerville Road, Wilmington, DE 19808; (302)636-3839, joan_stevens@agilent.com

Essential oils are used throughout many industries such as pharmaceuticals, cosmetics, aromatherapy and food. Residual levels of chemicals used in citrus crops are regulated because of their impact on public health and the environment. Another aspect is illegal “organic” product distribution. Organic product misrepresentation is a disturbing and serious problem which leads to consumers paying premium prices for non-organic product. It is believed that the pesticides are mainly located in the peel of the citrus fruit, considered as a protective layer. Essential oils are extracted from the peel. It takes several kilograms of extracted fruit peel to obtain several milliliters of the essential oils. The concentration of pesticides in the essential oil is thus much higher than in the fruit itself. This application describes a new, simple and rapid extraction procedure for the residues of pesticides in lemon and orange essential oils. The method involves the QuEChERS extraction and a modified dispersive SPE with analysis by GC/MS. Implementation of the QuEChERS extraction methodology and modified dispersive SPE enabled analysis of pesticides at less than 50 ppb and RSD’s less than 5% on average for the pesticides analyzed.

P-58b GC-µECD Analysis and Confirmation of CLP Pesticides in Olive Oil

Joan M. Stevens; **Agilent Technologies**, Department of Consumables and Supplies, 2850 Centerville Road, Wilmington, DE 19808; (302)636-3839, joan_stevens@agilent.com

An olive oil sample was analyzed for 20 contract laboratory protocol (CLP) pesticides. A QuEChERS extraction with dSPE cleaned the sample prior to analysis. A dual µECD and dual capillary GC column approach accomplished simultaneous primary and confirmatory analysis. The primary column, DB-35ms 30m x 0.25 mm x 0.25 µm and confirmatory column DB-XLB 30 m x 0.25 mm x 0.50 µm effectively resolve all 20 CLP pesticides with sensitivity in the low ppm range. An un-purged two-way capillary flow technology splitter divided the flow from a single injection port to the two analytical GC columns. Endosulfan sulfate and endosulfan I residues are confirmed to be present in the olive oil sample. In previous studies of pesticides residue analysis in olive oil, extraction procedures varied from LLE/SPE, GPC, and more recently to the use of QuEChERS methodology. QuEChERS extraction followed by dSPE is a convenient way to clean up sample matrixes just enough to remove chromatographic interferences and at the same time retain sensitivity for analytes of interest.

P-59a Increasing Efficiency and Pesticide Recovery from the QuEChERS Approach for Fruits and Vegetable Products using Ceramic Homogenizers

Joan M. Stevens; **Agilent Technologies**, Department of Consumables and Supplies, 2850 Centerville Road, Wilmington, DE 19808; (302)636-3839, joan_stevens@agilent.com

The QuEChERS approach is a methodology for the extraction of multiresidue pesticides/compounds from food matrices. Recovery and consistency for the compounds being extracted are directly affected by the degree of sample mixing and surface interaction with the salt buffers in the extraction step and with the dispersive SPE step. Inefficient shaking leads to lower recoveries and higher RSDs. Employing ceramic homogenizers during the QuEChERS extraction procedure compensated for variations in shaking technique. Recovery and efficiency were evaluated by LC/MS/MS at 10 second intervals throughout the 1 minute required manual shake time. The ceramic homogenizers drastically decreased the shaking time from 60 seconds to approximately 20 seconds, a 70% times savings. Maximum recoveries for several groups of pesticides were achieved within 20 seconds. The shape, angled cut and ceramic composition of the homogenizers all contribute to its ability to increase and facilitate the extraction of compounds. The ceramic homogenizers reduce variance between people and shaking techniques improving recoveries and RSDs for compounds being extracted by the QuEChERS methodology.

P-59b Determination of Quinolone Antibiotics in Bovine Liver using QuEChERS Extraction with Analysis by LC/MS/MS

Joan M. Stevens; **Agilent Technologies**, Department of Consumables and Supplies, 2850 Centerville Road, Wilmington, DE 19808; (302)636-3839, joan_stevens@agilent.com

Quinolones are a family of synthetic broad-spectrum antibiotics. There is evidence that quinolones in food animals leads to the emergence of quinolone-resistant bacteria. The resistant organisms are transmitted to humans via direct contact with the animal or through the consumption of contaminated food and water. Therefore, public health agencies in many countries, have established maximum residue limits (MRLs) of veterinary drugs in food-producing animals. Given the different drugs in different food origins and in different countries, the MRLs of quinolones in foodstuffs of animal origin are usually at the level of 100 µg/kg or higher. Employing the QuEChERS methodology has significant advantages over traditional methods; high recoveries for a wide range of pesticides, high sample throughput, labor and time saving, small solvent usage and waste, and manually accommodating; which has made QuEChERS a very popular methodology in recent years. Based on the chemical properties of the compounds of interest and food matrices, some modifications of the original method might be necessary to obtain accurate and precise results. The purpose of this work is to extend the QuEChERS methodology to veterinary drug residues in food producing animals.

P-60 LC-APPI-MS/MS for Trace Analysis of "Difficult" Pesticides

Sheng-Suan (Victor) Cai, Roland Carlson,[1] Karl Hanold, and Jack Syage; **Syagen Technology, Inc.**, 1411 Warner Ave, Suite D, Tustin, CA 92780; (714)727-0526, vcai@syagen.com
1. California Dept. of Food and Agriculture, Center for Analytical Chemistry, Meadowview Road, CA

APPI offers three major benefits for residue analysis: (1) Ionizing both polar and nonpolar compounds simultaneously; (2) Much reduced matrix effects than ESI; and (3) Over 4-5 orders of linear ranges. In this work, we demonstrate APPI sensitivity for analysis of 25 "difficult" pesticides included in the CDFA LC-ESI-MS/MS method, which requires acquisitions of $[M+NH_4]+$ adduct ions for ESI analysis. APPI analyses were performed on a Varian 320-MS/MS with APPI source. The calculated IDLs (P/P S/N=3) based on 250 pg FIA range from low femtogram to low picogram levels (see Table below). The ultimate goal of this research is to develop a comprehensive APPI based LC-MS/MS method to include those "difficult" compounds not amenable to ESI and previously exclusively analyzed by GC-MS.

P-61 APPI Enhanced DART Source for Ambient Screening

Jack A. Syage and Kaveh Jorabchi; **Syagen Technology, Inc**., 1411 Warner Ave., Tustin, CA 92780; (714)727-0522, jsyage@syagen.com

Direct Analysis in Real Time (DART) has become a widely adopted method to screen for compounds on a variety of surfaces and is proving a powerful means for achieving non-preparative high throughput analysis. We have investigated the complementary role that atmospheric pressure photoionization (APPI) can play in DART analysis. An APPI source was mounted orthogonally to the DART gas flow and inlet to the mass spectrometer. A low flow dopant delivery line (about 5 uL/min) was positioned in the DART gas flow just before the APPI source. About 10 test compounds were used spanning a range of non-polar (anthracene) to slightly polar (e.g., steroids) to polar (e.g., verapamil). 1 uL sample solutions in methanol were introduced by a capillary insertion probe as is commonly done with DART. The DART source was operated in both He and N2 discharge modes and was mated to an Agilent single quadrupole MS system. In positive mode the APPI source significantly enhanced signal for all compounds relative to DART alone. The signal enhancement in positive mode was dramatic for non-polar compounds such as anthracene and for more polar compounds was a factor of 3-5 in He mode and over an order of magnitude in N2 mode. Signal enhancement was also observed in negative mode. In summary APPI/DART

has the following advantages compared to DART alone: (i) expanded range of detectable compounds, (ii) enhanced sensitivity for all compounds, (iii) potential reduction in matrix suppression effects.

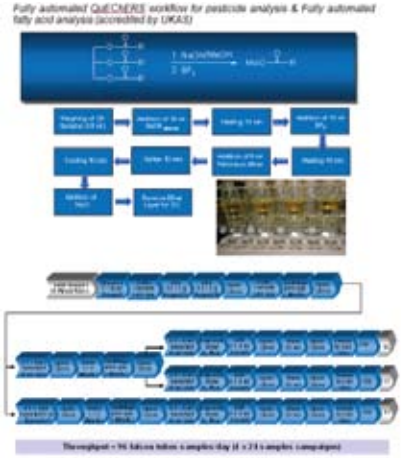
P-62 Pesticide Multiresidue Analysis in Tea Samples using Modified QuEChERS Method Combined with GC-NCIMS and LC-MS/MS

Xian Zhang, Jiugen Zhang, Nicole Mobley, Xiaomin Zheng, Oscar Ragin, Ling Lu, and Christopher Smith; **The Coca-Cola Company**, Department of Ingredient Quality, 1 Coca-Cola Plaza, Atlanta, GA 30313; (404)616-2382, xianzhang@na.ko.com

Tea, the agricultural product of the Camellia sinensis plant, is the most widely consumed beverage after water. Various pesticides are applied during different cultivation and storage stages. The resulting agricultural residues (ARs) are public concerns for potential health and environmental reasons. Therefore, the maximum residue limit (MRL) of pesticides has been regulated by many countries and international organizations. The MRLs are frequently modified to include more ARs and lower tolerance levels. Matrix interference presents significant challenges for analysis of ARs in tea samples. Because of the pigments, lipids, alkaloids, and other components in the samples, usually complicated and time-consuming extraction procedures are required. We have developed an improved protocol based on QuEChERS for the analysis of ARs in tea sample, which is quick and sensitive for extraction and quantification of 68 ARs. The new method yields low LODs, good recovery rates, and wide linear ranges. It is low-cost, reliable, and fast to meet the requirements of daily operations. We have successfully adopted the new method to analyze ARs in various commercial tea samples available on the market.

P-63 Accelerating Analytical Sample Preparation: Faster and Better by Flexible and Modular Automated Solutions

Jon Cartlidge and Michael Schneider;[1] **Chemspeed Technologies Inc.**, 113 North Center Dr, North Brunswick, NJ; (614)824-5962, jon.cartlidge@chemspeed.com
1. Chemspeed Technologies AG, Augst, Switzerland



Equipment similar to that which has originally been designed to accelerate Drug Discovery are now commonly being used for parallel, unattended Sample Preparation, Pretreatment, Digestion and Analysis. Multiple dispensing of solid and liquid samples, sample treatment with corrosive chemicals under harsh conditions, work-up steps like filtration, centrifugation & SPE purification are only some of the relevant workflow steps. Other technical requirements to these instruments might strongly differ from workflow to workflow. Generally a diverse set of complex unit operations, characteristic to each individual workflow, needs to be automated. Only flexible, modular equipment has the promise of covering the complex automation needs for faster and better analytical sample preparation. Using a selection of case studies, this presentation shows how a variety of challenging parallel sample preparation workflows has been fully automated.

P-64 Pesticide Levels in Wines from the Niagara Region of Canada

Dorothea F.K. Rawn, Sue C. Quade, and Wing-Fung Sun; **Health Canada, Food Research Division**, Sir Frederick Banting Research Centre, 251 Sir Frederick Banting Driveway, Tunney's Pasture PL 2203D, Ottawa, ON, K1A 0K9, **Canada**; 613-941-8958, sue.quade@hc-sc.gc.ca

Although most organochlorine insecticides have not been registered for use in Canada since the 1980s, endosulfan continues to be used on a variety of crops, including grapes, for the control of grape phylloxera, in the province of Ontario. Owing to its use on grapes in this region of Canada, Ontario wines were sampled in 2007 for endosulfan analysis. In general, 4 different wines were collected from each of the twenty-five wineries sampled (n=99). Individual wine samples were extracted using acetone:hexane (2:1) and cleaned up using gel permeation chromatography and Florisil. Analyses were performed using gas chromatography-high resolution mass spectrometry. Endosulfan I, II, and endosulfan sulfate levels in wines from Ontario were frequently below the limit of detection. The detection frequency of endosulfan (sum of I, II and sulfate) was 71% and when detected, concentrations ranged from (0.001 – 0.028 ng/g). The levels observed in the present study were well below the maximum residue limit (MRL) (1,000 ng/g) for total endosulfan allowed on raw grapes. Although endosulfan levels were very low in these Canadian wines, iprodione was detected at levels ranging from 0.28 ng/g to 300 ng/g, which is below the MRL for this fungicide in raw grapes and wine (10,000 ng/g and 5,000 ng/g, respectively).

P-65 Evaluation of a QUECHERS Type Extraction and Cleanup for Tandem LC-MS Determination of Veterinary Drugs and Other Contaminants in Tissue Samples

Michael S. Young, Jeremy C. Shia, and John Martin; **Waters Corporation**, 34 Maple Street, Milford, MA 01757; (508)883-7537, michael_s_young@waters.com

In recent years, a simple extraction and cleanup procedure (QUECHERS) has become accepted worldwide for the determination of pesticide residues in fruits and vegetables. In this presentation we will discuss modified QUECHERS procedures applied to the LC-MS determination of veterinary drugs in animal tissue. The purpose of this study is to evaluate methodologies for multi-class recovery of

antibacterial drugs. Among the antibacterial classes discussed will be the aminoglycoside, macrolide, fluoroquinolone, sulfonamide and beta-lactam. Among the topics discussed will be procedures for recovery of the highly water soluble compounds, and SPE clean-up options suitable for multi-class analysis.

P-66a Determination of Pesticides in Wine by GC-MS: ITD-EI-MS, SQ-NCI-SIM and TQ-EI-MS/MS

A. de Kok, I.R. Pizzutti,[1] L. Righi,[1] M. de Kroon, W. Wind, C.D. Cardoso,[1] R.C. Da Silva,[1] and J. Scholten; **Food and Consumer Product Safety Authority (VWA) –** National Reference Laboratory for Pesticides and Mycotoxins Analysis in Food, Amsterdam, **The Netherlands**; andre.de.kok@vwa.nl
1. Federal University of Santa Maria, CEPARC, Santa Maria, RS, **Brazil**

During this study various clean-up materials were tested to develop a new multi-method for simultaneous analysis of 112 pesticides (GC) and mycotoxins. A mixture of wine and water was extracted with 10 mL acetonitrile/1% acetic acid, followed by partitioning and buffering steps with sodium acetate. The validated clean-up procedure was done with PSA adsorbent, and wine extracts were measured by GC with different mass spectrometric detection modes, such as ITD-EI-MS, SQ-NCI-SIM and TQ-EI-MS/MS. For the linearity study, analytical curves for each pesticide were constructed with seven concentration from 0.4 to 200 $ngmL^{-1}$ (n=6) (TQ-EI-MS/MS and SQ-NCI-SIM), and from 4 to 400 ng mL^{-1} for ITD-EI-MS, comparing standard solutions in wine extract and in acetonitrile. For the recovery study, blank matrix was spiked at 5,10 and 25 $\mu g\ kg^{-1}$ for analysis by TQ-EI-MS/MS and SQ-NCI-SIM, and 10, 25 and 50 $\mu g\ kg^{-1}$ (n=6) for ITD-EI-MS. Using SQ-NCI-SIM 53 pesticides were analyzed, which 62% showed recoveries between 70-120% and RSD<20%, being successfully validated at the lowest level (5 $\mu g\ kg^{-1}$); 11% of the pesticides were validated with LOQ of 10 $\mu g\ kg^{-1}$ and 6% at 25 $\mu g\ kg^{-1}$. Some matrix effect was detected.

P-66b Simultaneous Determination of Pesticides and Mycotoxins in Wine by UPLC-MS/MS (ESI+)

I.R. Pizzutti, A. de Kok,[1] L. Righi, J. Scholten,[1] M. de Kroon,[1] W. Wind,[1] C.D. Cardoso, and R.C. da Silva; **Federal University of Santa Maria**, Center of Research and Analysis of Residues and Contaminants (CEPARC), Santa Maria, RS, **Brazil**; pizzutti@quimica.ufsm.br
1. Food and Consumer Product Safety Authority (VWA), Amsterdam, **The Netherlands**

Various tests with different adsorbents clean-up were done to develop a multi-method for simultaneous analysis of 143 pesticides and 33 mycotoxins in wine. It was diluted with water in different proportions and was extracted with acetonitrile/1% acetic acid, followed by a partitioning. The second drying is essential to correct problems about phase separation, due to the ethanol presence. For validation, pesticides were spiked at 5, 10 and 25 μgkg^{-1} and were analyzed six times at each spike level, under repeatability conditions. Linearity, recoveries, precision and matrix effects were determined. The mycotoxins were divided in 3 groups. The first containing Aflatoxin B1,B2,G1,G2 and Ochratoxin A were detectable more sensitively, and therefore spiked at 1, 2.5 and 10 μgkg^{-1}. The less sensitive ones (22 mycotoxins) were spiked at 50, 125 and 500 μgkg^{-1} and the last 6 mycotoxins were spiked at 10, 25 and 100 μgkg^{-1}. From the 143 pesticides, 72.5% showed results with acceptable recovery and RSD% and were successfully validated at the lowest spike level (5 μgkg^{-1}); 16.8% at 10 μgkg^{-1} and 4.6% at 25 μgkg^{-1}. The developed method has been applied successfully for a survey of pesticides and mycotoxins. Ochratoxin, showed recoveries between 91-98% with RSD between 11.8-23.7%.

P-67a Application of GC-MS (ITD-EI and SQ-NCI-SIM) and GC-MS/MS (TQ-EI) for the Development and Validation of a Multi-Residue Method for Pesticide Analysis in Breakfast Cereals

A. de Kok, I.R. Pizzutti,[1] G.N. Rohers,[1] M. de Kroon, W. Wind, C.D. Cardoso,[1] R.C. da Silva,[1] and J. Scholten
Food and Consumer Product Safety Authority (VWA) – National Reference Laboratory for Pesticides and Mycotoxins Analysis in Food, Amsterdam, **The Netherlands**; andre.de.kok@vwa.nl
1. Federal University of Santa Maria, CEPARC, Santa Maria, RS, **Brazil**

For the linearity study, analytical curves were constructed from 0.2 to 100 $ngmL^{-1}$ (7 concentrations) (n=6) for GC-MS(TQ-EI) and GC-MS(SQ-NCI-SIM), and from 2 to 200 $ngmL^{-1}$ for GC-MS(ITD-EI), comparing standard solutions in blank matrix and in acetonitrile. For the recovery study, blank cereal breakfast slurry was spiked at 10, 20 and 50 μgkg^{-1} for GC-MS(TQ-EI) and GC-MS (SQ-NCI-SIM), and 20, 50, 100 μgkg^{-1} (n=6) for GC-MS(ITD-EI). The extracts were measured by ITD-EI-MS, SQ-NCI-SIM-MS and TQ-EI-MS/MS modes. Various clean-up materials were tested and C18 was the most appropriate adsorbent. From the 51 pesticides analyzed by GC-MS(SQ-NCI-SIM), 59% showed recoveries between 70-120% and 62% had RSD ≤ 20%. The LOQ was 10 μgkg^{-1} for 63% of the pesticides and 50 μgkg^{-1} for 12%. The matrix effect was high for all analyzed pesticides. One of the main reasons that the target LOQ of 10 μgkg^{-1} could not always be achieved was the relatively unfavorable slurry ratio of 1:5 (matrix:water) that had to be used. Keeping the slurry/acetonitrile ratio constant (1:1), leads to a lower sample amount (2 g) and thus lower extract concentration, which results in a proportional higher LOQ. The presented method was reliable and robust enough for routine breakfast cereals analysis.

P-67b Application of UPLC-MS/MS (ESI+) for the Development and Validation of a Multi-Residue Method for Pesticide and Mycotoxin Analysis in Breakfast Cereals

I.R. Pizzutti, A. de Kok,[1] G.N. Rohers, J. Scholten,[1] M. de Kroon,[1] W. Wind,[1] C.D. Cardoso, R.C. da Silva;
Federal University of Santa Maria, Center of Research and Analysis of Residues and Contaminants (CEPARC), Santa Maria, RS, **Brazil**; pizzutti@quimica.ufsm.br
1. Food and Consumer Product Safety Authority (VWA), Amsterdam, **The Netherlands**

The goal was the development of a comprehensive and integrated method of analysis for 134 pesticides and 36 mycotoxins in corn flakes. Two different methods were compared by their extraction efficiency, for pesticides and mycotoxins. Method 1 used acetonitrile/1% acetic acid, followed by partitioning, without clean-up. Good recoveries were obtained for pesticides and most of the mycotoxins. Fumonisins showed low recoveries and for some mycotoxins matrix effect were significant. Method 2 used acetonitrile/4% formic acid, $MgSO_4$ and ammonium formate. Nexus was applied for dispersive SPE with extra amount of $MgSO_4$. Using Method 1, at 50 μgkg^{-1}, all pesticides (except dichlofluanide) showed recoveries between 70-120% and 90% with RSD<20%. Using Method 2, at the same level, 100% of the 134 pesticides showed recoveries in the acceptable range and only 22 pesticides with RSD>20%. Mycotoxins recoveries were also better applying Method 2. The clean-up showed a positive effect on the mycotoxins results, as to recoveries, RSD and lower matrix effect, but not on the pesticides. Many pesticides showed results outside the acceptable range at the 20 and 10 μgkg^{-1} because the relatively low sample amount (2 g) was taken for the sample extraction. Selectivity, accuracy, repeatability, linearity, LOD, LOQ were determined.

P-68a Multi-Method Development and Validation for Pesticide Analysis in Green Coffee Beans using GC-MS/MS (TQ-EI), GC-MS (SQ-NCI-SIM and ITD-EI)

A. de Kok, I.R. Pizzutti,[1] B. Reichert,[1] M. de Kroon, W. Wind, C.D. Cardoso,[1] R.C. da Silva,[1] J. Scholten;
Food and Consumer Product Safety Authority (VWA) – National Reference Laboratory for Pesticides and Mycotoxins Analysis in Food, Amsterdam, **The Netherlands**; andre.de.kok@vwa.nl
1. Federal University of Santa Maria, CEPARC, Santa Maria, RS, **Brazil**

Brazil has experienced serious export problems to Japan due to findings of pesticide residues in coffee, which exceeded their MRLs. Due to the increase of pesticides applied in coffee a stricter control of the residues is required. This project aimed to optimize and validate an acetonitrile-based extraction method for a large number of pesticides and mycotoxins in green coffee beans. A slurry was prepared of green milled coffee and water which was then extracted with acetonitrile/acetic acid, followed by $MgSO_4$ addition. Various clean-up adsorbents were tested but the best results were obtained using 500mg of C18. The validation was performed by analyzing fortified samples at 10, 20 and 50 μgkg^{-1}, 6 replicates at each level. Three different detection systems/modes were used: GC-MS/MS (TQ-EI), GC-MS (SQ-NCI-SIM) and GC-MS (ITD-EI). Linearity, accuracy, precision and matrix effect were determined for each individual pesticide. From the 58 pesticides analyzed by GC-MS (SQ-NCI-SIM), 75% had recoveries between 70-120% and 69% had RSD≤20%. The method LOQ was 10 μgkg^{-1} for 60%, 20 μgkg^{-1} for 5%, and 50 μgkg^{-1} for 14% of the pesticides, respectively. The matrix effects were very high for all analyzed pesticides, which means that matrix-matched calibration is a prerequisite for reliable quantification.

P-68b Multi-Method Development and Validation for 134 Pesticides and 36 Mycotoxins Analysis in Green Coffee Beans using UPLC-MS/MS (ESI+)

I.R. Pizzutti, A. de Kok,[1] B. Reichert, J. Scholten,[1] M. de Kroon,[1] W. Wind,[1] C.D. Cardoso, R.C. da Silva;
Federal University of Santa Maria, Center of Research and Analysis of Residues and Contaminants (CEPARC), Santa Maria, RS, **Brazil**; pizzutti@quimica.ufsm.br
1. Food and Consumer Product Safety Authority (VWA), Amsterdam, **The Netherlands**

The purpose was to develop and validate a method able to identify and quantify a large number of pesticide residues and also mycotoxins in green coffee beans. Several clean-up were tested in combination with a modified QuEChERS extraction. A slurry of green milled coffee and water was prepared and then extracted with acetonitrile/acetic acid, followed by phase partitioning. The pesticides were spiked at 10, 20 and 50 μgkg^{-1} and analyzed with a UPLC-MS/MS ESI^+, under repeatability conditions, 6 replicates (n=6) at each spike level. Linearity, accuracy, precision, matrix effects and Ion ratios (identification purposes) were validated and compared with guidance criteria. More than 90% of the pesticides obtained average recoveries between 70−120%. The RSD was ≤ 20% for more than 84% of the pesticides. From the 134 pesticides analyzed, for 61% a LOQ of 10 μgkg^{-1} was achieved, for 19% of 20 μgkg^{-1}, and for 9% of 50 μgkg^{-1}. Matrix effects were significant for 16% of the pesticides, but had no considerable influence on the obtained validation data. The results obtained during the validation show that the method is appropriate to determine residues of pesticides and mycotoxins in green coffee, because the data are satisfactory according to SANCO 2009.

NOTES

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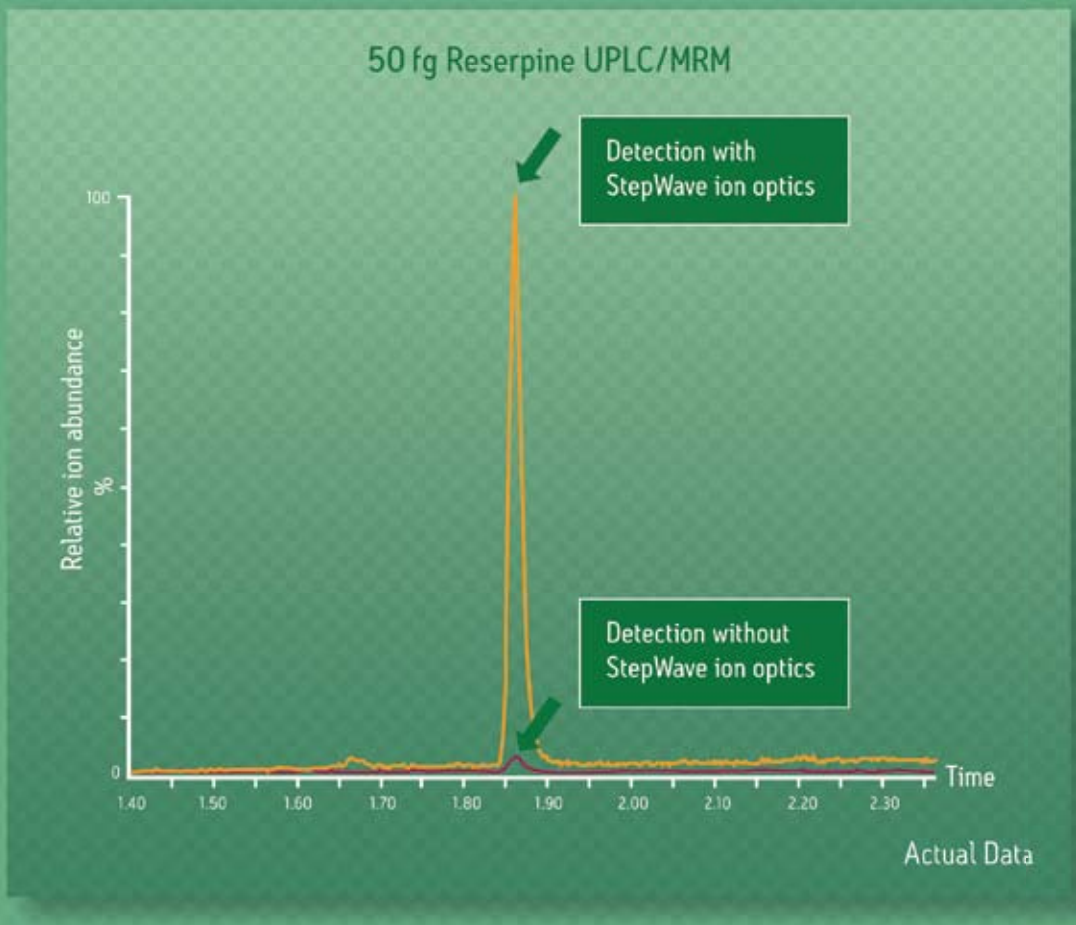
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