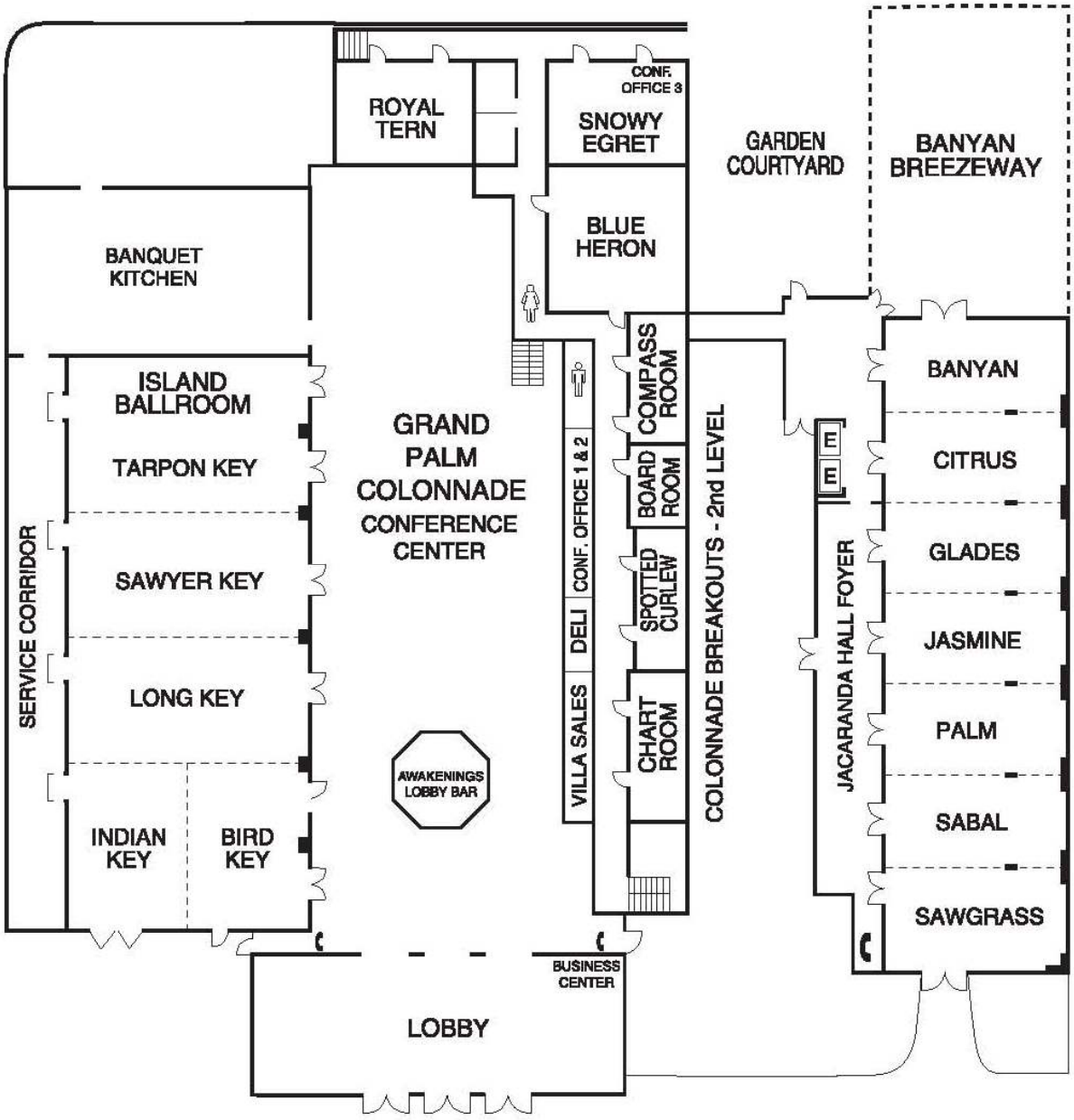
Conference Center Maps



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

Exhibits, Posters, Reception: Banyan Breezeway (adjacent to Courtyard)

Vendor Seminars: Banyan & Citrus Rooms (across the hall from exhibits)

MEETING AT-A-GLANCE

Sunday Evening, July 20, 2008

5:00 - 8:00 pm	Registration	Grand Palm Colonnade
6:00 - 7:30 pm	**Restek Evening Seminar**	Banyan & Citrus Rooms (Jacaranda Hall)
6:00 pm	Exhibition Set Up Begins	Banyan Breezeway
8:00 pm	Poster Set Up Begins	Banyan Breezeway
6:00 - 10:00 pm	Technical Session Setup	Long / Bird / Indian Key Ballrooms
8:00 - 8:30 pm	Moderator Training	Long / Bird / Indian Key Ballrooms

Monday, July 21, 2008

All Day	Exhibition	Banyan Breezeway
All Day	**Session "A" Posters**	Banyan Breezeway
7:30 - 8:30 am	Registration	Grand Palm Colonnade
7:30 - 8:30 am	Early Morning Coffee	Banyan Breezeway
7:30 - 8:15 am	**Agilent Breakfast Seminar**	Banyan & Citrus Rooms (Jacaranda Hall)
8:30 – 12:00 noon	**FPRW Technical Session - Emerging Residue Issues**	Long / Bird / Indian Key Ballrooms
12:15 - 1:15 pm	**Waters Luncheon Seminar**	Banyan & Citrus Rooms (Jacaranda Hall)
1:30 - 4:30 pm	**FPRW Technical Session - QuEChERS & its Variations**	Long / Bird / Indian Key Ballrooms
4:30 - 5:30 pm	**QuEChERS Open Forum**	Long / Bird / Indian Key Ballrooms
6:30 - 7:30 pm	**Evening Reception & Poster Session "A"**	Banyan Breezeway

Tuesday, July 22, 2008

All Day	Exhibition	Banyan Breezeway
All Day	**Session "B" Posters**	Banyan Breezeway
7:30 - 8:30 am	Registration	Grand Palm Colonnade
7:30 - 8:30 am	Early Morning Coffee	Banyan Breezeway
7:30 - 8:15 am	**ThermoFisher Scientific Breakfast Seminar**	Banyan & Citrus Rooms (Jacaranda Hall)
8:30 - 10:25 am	**FPRW Technical Session - Instrumental Technologies**	Long / Bird / Indian Key Ballrooms
11:00 - 12:00 noon	**Poster Session "B"**	Banyan Breezeway
12:15 - 1:15 pm	**Applied Biosystems Seminar**	Banyan & Citrus Rooms (Jacaranda Hall)
1:30 - 3:15 pm	**FPRW Technical Session - Emerging Pesticide Issues**	Long / Bird / Indian Key Ballrooms
4:00 - 5:00 pm	**MS Users Meeting**	Long / Bird / Indian Key Ballrooms
5:00	Beach Volleyball	On the Beach
5:30 - 6:30 pm	**Organizing Committee Meeting**	Long / Bird / Indian Key Ballrooms

Wednesday, July 23, 2008

7:30 - 8:30 am	Registration	Grand Palm Colonnade
7:30 - 8:30 am	Early Morning Coffee	Grand Palm Colonnade
7:30 - 8:15 am	**Agilent Breakfast Seminar**	Banyan & Citrus Rooms (Jacaranda Hall)
8:30 - 12:00 noon	**FPRW Technical Session - Theory and Practice**	Long / Bird / Indian Key Ballrooms
12:15 - 1:15 pm	**Varian, Inc. Luncheon Seminar**	Banyan & Citrus Rooms (Jacaranda Hall)

GENERAL INFORMATION

Registration

Check in once at the registration desk at your earliest opportunity

Sunday - 5:00-8:00 PM
Monday - 7:30-8:30 AM
Tuesday - 7:30-8:30 AM
Wednesday - 7:30-8:30 AM

KEY to Presentation Numbering System

FPRW talks are numbered **1, 2, 3 …**
FPRW Session "A" posters are numbered, **A1, A2, A3 …**
FPRW Session "B" posters are numbered, **B1, B2, B3 …**
Presenters' addresses are cross-indexed to these numbers.

Poster Session

Hang Posters for Session "A" on Sunday evening, beginning at 8:00 pm.
Take down Session "A" posters immediately following the reception.
Hang Posters for Session "B" immediately following the reception on Monday evening or Tuesday morning, beginning at 7:30 am.
Take down Session "B" posters on Tuesday, 5:00 pm.
Posters may be viewed at any time, but authors will be at their posters for Poster Session "A" during the reception on Monday evening and for Poster Session "B" on Tuesday, 11:00 - 12:00 noon.

Exhibition

Monday - 7:30 am - 5:00 pm, and during Monday evening reception 6:30-7:30 pm
Tuesday - 7:30 am - 5:00 pm

Coffee and Breaks

Coffee will be available 7:30 - 8:30 am each morning. There will be morning refreshment breaks each day and afternoon refreshment breaks on Monday and Tuesday. All Monday and Tuesday breaks will be served in the Banyan Breezeway in conjunction with the Exhibition. On Wednesday the morning breaks will be served in the Grand Palm Colonnade.

Announcements

Moderators will make general announcements from the podium. If you need to convey such information to a moderator, you must fill out an announcement form and submit it to W. George Fong. These announcement forms will be available at the registration desk.

Door Prizes

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

Job Placement Bulletin Board

Self serve message board for those offering or seeking employment

Abstracts

There are brief abstracts published for the FPRW meeting. However, you can also contact the authors using the contact information in the program. With presenter permission, we publish speaker presentations on our website following the meeting.

Copies of Presentations

Oral Presentations: Following the meeting, if time and resources permit, some of the oral presentations will be posted on our website. 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. If this does not meet your need, then please contact the speaker directly.

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

Attendee List

After the meeting (approx 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 FPRW DATES

2009 July 19 - 22 TradeWinds Island Grand Resort, St. Pete Beach, Florida

Add www.FLworkshop.com to your browser favorites

MODERATORS and PROGRAM COMMITTEE

(C=PROGRAM COMMITTEE, M=MODERATOR)

M, C, Garris, Sherry T, SC Dept. of Agriculture, 1101 Williams Street, Columbia, SC, USA 29201, *sgarris@scda.sc.gov*
M, C, Hernandez, Amy, LSU Agricultural Center, Rm102 AGCHEM BLDG LSU CAMPUS, Baton Rouge, LA, USA 70803, *amy_h@ldaf.state.la.us*
M, C, Krynitsky, Alex, FDA-CSAN, 5100 Paint Branch Parkway; HFS-717, College Park, MD, USA , *alex.krynitsky@fda.hhs.gov*
M, C, LeCompte, Danny, Alabama Pesticide Residue Laboratory, 1081 South Donahue Drive, Auburn, AL, USA 608-829, *lecomod@auburn.edu*
M, C, McMullen, Sarah, FDA, 60 Eighth St NE, Atlanta, GA, USA 30309, *sarah.mcmullen@fda.hhs.gov*

C, Brown, Amy, Florida Dept. of Agriculture, 3125 Conner Blvd. Lab 3, Tallahassee, FL, USA 32399, *browna@doacs.state.fl.us*
C, Cook, Jo Marie, Florida Department of Agriculture and Consumer Ser, 3125 Conner Blvd. Lab #3, Tallahassee, FL, USA 32399, *cookj@doacs.state.fl.us*
C, Eigen, Susan, Georgia Department of Agriculture, P. O. Box 1507, Tifton, GA, USA 31793-1507, *seigen@agr.state.ga.us*
C, Eitzer, Brian, The Conn. Agr. Exp. Station, 123 Huntington St., New Haven, CT, USA , *brian.eitzer@po.state.ct.us*
C, Farrow, Michael, Georgia Department of Agriculture, P. O. Box 1507, Tifton, GA, USA 31793-1507, *mfarrow@agr.state.ga.us*
C, Fourrier, Loretta, LSU Agricultural Center,Rm 102 AGCHEM BLDG LSU CAMPUS, Baton Rouge, LA, USA 70803, *loretta@ldaf.state.la.us*
C, Hammack, Walter, Florida Dept. of Agriculture, 3125 Conner Blvd Chemical Residue Lab #3, Tallahassee, FL, US , *hammacw@doacs.state.fl.us*
C, Huffstetler, Nina, LSU Agricultural Center, Rm102 AGCHEM BLDG LSU CAMPUS, Baton Rouge, LA, USA 70803, *nina@ldaf.state.la.us*
C, Krol, Walter, The Conn. Agr. Exp. Station, 123 Huntington St., New Haven, CT, USA , *walter.krol@po.state.ct.us*
C, Lucas, Patty, Florida Dept. of Agriculture, 3125 Conner Blvd. Lab 5, Tallahassee, FL, USA 32399, *lucasp@doacs.state.fl.us*
C, Meiers, Rebecca, Florida Dept. of Agriculture, 3125 Conner Blvd. Lab 3, Tallahassee, FL, USA 32399, *meiersr@doacs.state.fl.us*
C, Parker, Gail, Florida Dept. of Agriculture, 3125 Conner Blvd. Lab 3, Tallahassee, FL, USA 32399, *parkerg@doacs.state.fl.us*
C, Rygiel, Teresa, Florida Dept. of Agriculture, 3125 Conner Blvd. Lab 5, Tallahassee, FL, USA 32399, *rygielt@doacs.state.fl.us*

Jo Marie Cook, 2008 FPRW Program Chair

Alex Krynitsky, 2009 FPRW Program Chair

2008 EXHIBITORS

Exhibition Location: Banyan Breezeway

In alphabetical order

ABC Laboratories, Inc.	www.abclabs.com
AccuStandard, Inc	www.accustandard.com
Adpen Laboratory	www.adpen.com
Agilent Technologies	www.agilent.com/chem
Applied Biosystems	www.appliedbiosystems.com
Chromsys LLC	www.chromsys.com
Dionex Corporation	www.dionex.com
Gerstel	www.gerstelus.com
Gilson, Inc.	www.gilson.com
Horizon Technology, Inc.	www.horizontechinc.com
LECO Corporation	www.leco.com
OI Analytical	www.oico.com
Perkin Elmer Corp.	www.perkinelmer.com
Pickering Laboratories Inc	www.pickeringlabs.com
Quantum Analytics	www.lqa.com
Resolution Analytical Systems LLC	www.resolutionsys.com
Restek	www.restek.com
SCI-CON	www.scicon.net
SPEX CertiPrep Group	www.spexcsp.com
Thermo Fisher Scientific	www.thermo.com/ms
UCT	www.unitedchem.com
Varian	www.varianinc.com
Waters Corporation	www.waters.com

SEE PAGE 7 & 8 for FREE EXHIBITOR SEMINARS

2008 / 45th Florida Pesticide Residue Workshop

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

Vendor Seminars: Please sign up at the meeting registration desk

1. Sunday Evening, July 20, 2008

6:00 - 7:30 pm **Restek**

Comprehensively Defining the Dispersive SPE of QuEChERS Samples with GC-MS

Mr. Jack Cochran

The QuEChERS (quick, easy, cheap, effective, rugged, and safe) method of preparing fruit and vegetable extracts for pesticide analysis uses a novel dispersive solid phase extraction (dSPE) step for extract cleanup. The primary sorbent is PSA, but other materials can also be employed, such as graphitized carbon black, C18, and aminopropyl. Often though these materials are employed without fully determining how they are performing to remove matrix components that might lead to quantitative bias, chromatographic overload and retention time shifts, or instrumental degradation. This presentation will look at a variety of dSPE procedures and evaluate them not only for pesticide recoveries, but also matrix component removal. The work is facilitated by using a sensitive, full mass range, GC-TOFMS system.

2. Monday Breakfast, July 21, 2008

7:30 - 8:15 am Agilent Technologies, Inc.

Multi-residue Pesticide Screening and Confirmation with Advances in LC/MS/MS

Jerry Zweigenbaum, Ph.D.

Sensitivity for many pesticides is very good in liquid phase tandem MS analysis. Yet, there are compounds that fall in that grey area of poor sensitivity and make it difficult to meet stringent requirements (such as those for baby foods). In this presentation we will describe advances in instrument design that improve overall sensitivity and thus make it easier to meet worldwide detection limits for pesticides in question and provides overall improvement in the multi-residue analysis. Also, there are limitations in the number of analytes that can accurately be monitored in one method with the use of selected reaction monitoring (SRM) techniques. Advances in this area and how they impact the analysis will be described. Although the use of triple quadrupole MS/MS is excellent for targeted analysis, it does not address the need for non-target analysis and the identification of unknowns. This subject will be discussed and a workflow to address this need will be described.

3. Monday Lunchtime, July 21, 2008

12:15 - 1:15 **Waters**

A rapid method for the determination and confirmation of over 400 pesticide residues in food

Brad Barrett and Kevin Jenkins

As there are currently well over 1,000 pesticides in use, laboratories are under increasing pressure to broaden the range of pesticides determined in a single analysis over a shortened run time. The need to meet mandated detection limits, develop sample preparation techniques for complex matrices and the desire to increase sample throughput are the main challenges facing food safety testing laboratories today. The use of a single multi-residue method per instrument can dramatically improve laboratory workflow. The screening of more than 400 pesticide residue compounds in fruit and vegetables was achieved using liquid chromatography combined with tandem quadrupole mass spectrometry (LC/MS/MS) operated in multiple reaction monitoring (MRM) mode. Using a generic d-SPE (dispersive solid phase extraction) procedure, valid for a wide range of compound classes in a representative set of food commodities, the single extract was injected twice using an Ultra Performance Liquid Chromatography (UPLC™) method of ten minutes with two MRM transitions per compound. The limits of determination achieved for the pesticides analyzed are well below that required for worldwide surveillance monitoring.

4. Tuesday Breakfast, July 22, 2008

7:30 - 8:15 am **Thermo Fisher Scientific**

Cradle to Grave Environmental & Food Safety Solutions using TSQ Quantum LC-MS Systems

Dipankar Ghosh, Ph.D

Achieving low LODs of pesticides, antibiotics and veterinary residues in drinking water and food substances is of paramount importance in order to achieve the regulatory levels. These substances pose a significant health threat

and, therefore, need to be accurately detected. Traditionally, LC-MS/MS has been used for the identification and quantification of these residues. However, this methodology typically requires extensive offline sample pre-concentration which can be particularly time consuming and expensive. Enhanced sensitivity & workflow optimization techniques for the analysis of ppt levels of pesticides, antibiotics, and pharmaceutical contaminants in water and food matrices using TSQ Quantum triple quadrupole mass spectrometers will be highlighted. These total online solutions coupled to a triple quadrupole mass spectrometer have the potential to significantly reduce sample preparation time from days to minutes, thus improving the productivity in high throughput laboratories.

Optimization of a Complete Analysis of Pesticide Residues in Produce by GC/MS
Eric Phillips

This presentation will cover the complete method development of the analysis of pesticide residues in produce. The QuEChERS extraction technique was used to prepare samples for analysis. A simple flow chart for this extraction will be provided. The method development procedures for chromatography and ion trap and triple quad mass spectrometers will be discussed. Some helpful hints and will be provided to ensure the analysis can be performed for a large number of samples over a long period of time without venting the systems

5. Tuesday Lunchtime, July 22, 2008
12:15 - 1:15 pm **Applied Biosystems**

Strategies for Rapid Pesticide Measurement and confirmation by LC/MS/MS
Lutz Alder, Federal Institute for Risk Assessment, Berlin, Germany

Nowadays, the tandem mass spectrometry has become an indispensable tool for pesticide residue analysis. After a short period of use as supplementary technique, LC-MS/MS instruments are now the workhorses in multi-residue pesticide analysis. Another aspect is the availability of several new analytical methods, which significantly reduces the time needed for sample preparation and clean-up. Subsequently, it is often easier to produce lots of final sample extracts than to measure them by HPLC combined with tandem mass spectrometry.

6. Wednesday Breakfast, July 23, 2008
7:30 - 8:15 am **Agilent**

Part 1: Previewing Agilent's GC Triple Quadrupole MS
Part 2: Comprehensive Pesticide Analysis at the 10 ppb level by GC/MS with Deconvolution Reporting Software"
Philip L. Wylie, Ph.D.

Complete pesticide residue analysis depends on broad screening in combination with target compound analysis. Comprehensive screening is accomplished in the scan mode using Agilent's 7890/5975C single quadrupole GC/MS with Deconvolution Reporting Software and the RTL Pesticide Library. Target pesticide analysis will benefit from the new Agilent 7000A triple quadrupole GC/MS/MS, which is based on the Agilent MSD. The 7000A has been designed from the ground up for routine, high-performance, high-throughput operation and is an ideal tool for low level pesticide analysis in complex matrices.

7. Wednesday Lunchtime, July 23, 2008
12:15 - 1:15 **Varian**

Increasing Lab Efficiency and Production via Multiple Pesticide Residue Analysis by GC/MS/MS and LC/MS/MS
Nik Hubbard

Learn how to save your lab time and money by taking advantage of the selectivity provided by both GC & LC triple quadrupole mass spectrometry. Many modern compounds which have better activity or less environmental impact are either not amenable to GC or are not detectable at sufficiently low levels by traditional GC or GC/MS methods in order to meet compliance with statutory maximum residue levels. More and more compounds therefore have to be tested separately by specific methods resulting in lengthy analysis times with high staff and consumable costs. This does not have to be the case. The selectivity, sensitivity and speed afforded by the use of modern triple quadrupole mass spectrometers enables a more generic approach to extraction with minimal clean up and provides individual methods for quantifying more than 200 compounds in a single analysis.

Monday Morning, July 21, 2008

7:30 - 8:15 Agilent Technologies Breakfast Seminar
Location: Banyan & Citrus Rooms (Jacaranda Hall)

ALL DAY **Poster Session "A", Banyan Breezeway**

Emerging Residue Issues

Location: Long / Bird / Indian Key Ballrooms

Moderator: Sarah McMullen, Food and Drug Administration

8:30 - 9:00 FPRW, FLAG Works and You!
J. D Warren, FLAG Works &
Jo Marie Cook, FL Dept. Agriculture & Consumer Services

1 9:00 - 9:45 Keynote: Matrix Effects in Multiresidue Liquid Chromatography/Electrospray Ionization Tandem Mass Spectrometric Analysis of Emerging Organic Pollutants
Paul Yang, Ontario Ministry of the Environment

9:45 - 10:30 **Break (w/ refreshments) - PLEASE VISIT EXHIBITORS**
Location: Banyan Breezeway

2 10:30 - 11:15 Implementing ISO 17025 Accreditation in Costa Rica Laboratories
Cristina Chinchilla and Mario Masis, Universidad De, Costa Rica

3 11:15 - 11:45 Two Case Studies: Validating Methods to Detect Inadvertently Released Grains into the Marketplace
Ron Jenkins, USDA

11:45 - 12:00 Door Prize Drawing - You must be present to win!

12:00 - 1:30 **Lunch** (on your own)

12:15 - 1:15 Waters Corporation Luncheon Seminar
Location: Banyan & Citrus Rooms (Jacaranda Hall)

Monday Afternoon, July 21, 2008

Location: Long / Bird / Indian Key Ballrooms

QuEChERS & Its Many Variations

Moderator: Amy Hernandez, Louisiana State University

4	1:30 - 1:55	Pesticide Multiresidue Analysis in Cereal Grains using Modified QuEChERS Method *Katerina Mastovska, USDA-ARS*
5	1:55 - 2:20	Pesticide Residue Analysis of Honey Bee Products Using Quechers *Roger Simonds, USDA*
6	2:20 - 2:45	A Modified QuEChERS Procedure for the UPLC-MS/MS Analysis of Pesticides in Fresh Produce *Jon Wong, FDA*
	2:45 - 3:30	**Break (w/ refreshments) - PLEASE VISIT EXHIBITORS** Location: Banyan Breezeway
7	3:30 - 3:55	Effect of Chlorophyll on Long-Term GC Performance in Pesticide Residue Analysis *Steven Lehotay, USDA-ARS*
8	3:55 - 4:20	OP Pesticides at ppb Levels using QuEChERS & GC-PFPD *Frank Schenck, FDA, Atlanta Regional Laboratory*
	4:20 - 4:30	Door Prize Drawing - You must be present to win!
9	4:30 - 5:30	**QuEChERS Open Forum:** Discuss method modifications and performance, new and challenging analytes, detection limits, etc *Moderator: Katerina Mastovska, USDA-ARS*
	6:30 - 7:30	**Reception and Poster Session "A"** Compliments of the TradeWinds Island Grand (Cash bar) Location: Banyan Breezeway

Tuesday Morning, July 22, 2008

Location: Long / Bird / Indian Key Ballrooms

7:30 - 8:15 Thermo Fisher Scientific Breakfast Seminar
Location: Banyan & Citrus Rooms (Jacaranda Hall)

ALL DAY **Poster Session "B", Banyan Breezeway**

Instrumental Technologies

Moderators: Alex Krynitsky, Food and Drug Administration

8:30 - 8:40 "Welcome"

10 8:40 - 8:55 Highlights of the European Pesticide Residue Workshop
Lutz Alder,
Federal Institute for Risk Assessment, Berlin, Germany

11 8:55 - 9:20 No More Trouble with Matrix Effects? A Simple Way to Consider Matrix Effects in ESI-MS without Standard Addition, Matrix Matched or Stable Isotope Labeled Standards
Helen Stahnke and Lutz Alder, Federal Institute for Risk Assessment, Berlin, Germany

12 9:20 - 9:55 Comprehensive Two-Dimensional Gas Chromatography/Time-of-Flight Mass Spectrometry: Advances and Perspectives for Environmental and Bioanalytical Screening
Jean-Marie Dimandja, Spelman College

13 9:55 - 10:20 Multi-residue Methods using LC/TOF-MS for the Analysis of Pesticides in Food
Imma Ferrer, University of Colorado

10:20 - 10:30 Door Prize Drawing - You must be present to win!

10:30 -11:00 **Break (w/ refreshments) - PLEASE VISIT EXHIBITORS**
Location: Banyan Breezeway

11:00 - 12:00 **Poster Session "B" - Authors available at their posters**
Location: Banyan Breezeway

12:00 - 1:30 **Lunch** (on your own)

12:15 - 1:15 Applied Biosystems Luncheon Seminar
Location: Banyan & Citrus Rooms (Jacaranda Hall)

Tuesday Afternoon, July 22, 2008

Location: Long / Bird / Indian Key Ballrooms

Emerging Pesticide Issues

Moderator: Sherry Garris, South Carolina Department of Agriculture

14 1:30 - 2:00 Biomonitoring of Exposure to Pesticides
Dana Barr, Centers for Disease Control

15 2:00 - 2:30 Ethanol Fuel: The Impact of Aflatoxin Degradation
William Holmes, Mississippi State University

16 2:30 - 3:00 Development and validation of a multi-residue method for the analysis of neonicotinoid and macrocyclic lactone pesticides in milk and 11 fruit and vegetable commodities by LC-MS/MS.
Alaa Kamel, U.S. EPA

3:00 - 3:15 Door Prize Drawing - You must be present to win!

3:15 - 4:00 **Break (w/ refreshments) - PLEASE VISIT EXHIBITORS**
Location: Banyan Breezeway

17 4:00 - 5:00 **MS Users Meeting**
Moderator: William Holmes, Mississippi State University
Share challenges and solutions: quantitation issues, ionization issues, matrix effects, ion suppression and enhancement, confirmation criteria, best sample introduction techniques, pros and cons of various analyzers (e.g. traps, quads, TOF, etc.)

5:00 Beach Volleyball

5:30 - 6:30 **Organizing Committee Meeting**
All interested attendees and exhibitors are invited to attend.

Wednesday Morning, July 23, 2008

Location: Long / Bird / Indian Key Ballrooms

7:30 - 8:15 Agilent Technologies Breakfast Seminar
Location: Banyan & Citrus Rooms (Jacaranda Hall)

Trace Level Analysis, Theory and Practice

Moderator: Danny LeCompte, Alabama Pesticide Residue Laboratory

18 8:30 - 9:00 Dissipation studies of pesticide residues in fruits and vegetables and human risk assessment,
Paula Paya, University of Murcia, Spain

19 9:00 - 9:30 Multi-Residue Pesticide Analysis on Incurred Produce Samples using Variation of Disposable Pipette Extraction
William Brewer, University of South Carolina

20 9:30 - 10:00 Comparison of HPLC- with UPLC-MS/MS Multiresidue Methods for the Determination of Pesticide Residues in Primary Agricultural Products: Extension of Scope (>220 pesticides) vs. Shorter Run Times
André de Kok, National Reference Laboratory for Pesticides Analysis, The Netherlands.

10:00 - 10:45 **Break (w/ refreshments)**
Location: International Ballroom Foyer

21 10:45 - 11:15 Methods for Extractable Nonvolatile Compounds Analyzed by Liquid Chromatography/Mass Spectrometry for Environmental Restoration Following Homeland Security Events
Lawrence Zintek, U.S. EPA

22 11:15 - 11:45 Recent Analytical Challenges: Proportionality Studies
Lynda Podhorniak, U.S. EPA-OPP

11:45 - 12:00 Door Prize Drawing - You must be present to win!

12:15 - 1:15 Varian Luncheon Seminar
Location: Banyan & Citrus Rooms (Jacaranda Hall)

Meeting Adjourns

FPRW, July 19 - 22, 2009, TradeWinds Island Grand Resort, St. Pete Beach

All Day Monday

Location: Banyan Breezeway

POSTER SESSION A

A-1 Schreiber, Andre, Applied Biosystems,
LC/MS/MS for the Quantitation and Confirmation of Endocrine Disrupting Compounds, Pharmaceuticals and Personal Care Products in Water Samples with Library Search Confirmation

A-2 Schreiber, Andre, Ozeki, Yuriko, Baker, CJ, Applied Biosystems
Using the MRM catalogue of Cliquid Software to Quickly Build Custom LC/MS/MS Methods for pesticide analysis matching the new Japanese Positive List

A-3 Schreiber, Andre, Caraiman, Doina, Pace, Nadia, Baker, CJ, Applied Biosystems
Accelerated LC/MS/MS for the Quantitation and Confirmation of Pesticides in Food and Water Samples

A-4 Nik Hubbard, Varian
Multi-Residue Analysis of Pesticides in Vegetable and Fruit Extracts by Gas Chromatography Mass Spectrometry

A-5 Beck, Jonathan, Thermo Fisher Scientific,
Comparing Large Injection Volumes and Online Pre-concentration in Water Analysis

A-6 Phillips, Eric, Thermo Fisher Scientific
Multi-residue Pesticide Analysis Using Ion Trap GC/MS/MS Analysis with a Modified QuEChERS Extraction for Lettuce

A-7 Beck, Jonathan, Thermo Fisher Scientific,
Increased Mass Resolution to Reduce Matrix Interference with the LC-MS/MS Analysis of Pesticides in Food Residues

A-8 Stevens, Joan, Gilson Inc**.,**
Determination of Veterinary Drug Residues in Fish by an Automated SPE-HPLC System

A-9 Stevens, Joan, Gilson Inc.,
The Examination and Automation of GPC, SPE and QuEChERS for Pesticides in Oil

A-10 Wylie, Phil , Agilent Technologies
Evaluating a Method to Analyze Pesticides at the 10 ppb Level Using GC/MS with Large Volume Injection, Deconvolution Reporting Software, and a Retention Time Locked Pesticide Library

A-11 Wylie, Phil, Agilent Technologies
Bridging the Performance Gap Between GC/MS and GC/MS/MS with Deconvolution Technology

A-12 Zweigenbaum, Jerry, Agilent Technologies
HILIC Separation of Herbicides and Other Quaternary Ammonium Compounds by LC/MS

A-13 Shofran, Brian, LECO Corporation
Rapid Analysis of Carbamate Pesticides using LC-TOFMS

A-14 Hilton, Donald, LECO Corp
Screening for Environmental Contaminants in Complex Matrices - Tobacco

A-15 Young, Michael, Waters Corporation
SPE Protocol For Rapid UPLC-MS Determination Of Acidic Pesticides

A-16 Watts, Vivian, Chromsys
Comparing SIM and MRM for pesticide analysis in a complex grain matrix

A-17 Settle, Virgil, Gerstel
Automation of Sample Preparation Steps Using a Robotic Autosampler

A-18 Sheldon Henderson, Dionex
ASE and Productivity Gains in Environmental Analyses

A-19 Johnny George, Horizon Technology, Inc
Automated Solid Phase Extractions of Organochlorine Pesticides from Water

A-20 Jack Cochran, Restek
A New Capillary Column for the Analysis of Pesticides by GC/MS

A-21 Robert Wohleb, Ian Wohleb and Marc Elliott, Resolution Systems LLC
Immobilized Liquid Extraction of Organic Residues from Aqueous Matrices as a "Low Solvent" Sample Preparation Technique for GC/LC Analysis

A-22 Laura Chambers, OI Analytical
Using the Pulsed Flame Photometric Detector (PFPD) for Low-level Analysis of Organophosphorus Pesticides

All Day Tuesday

Location: Banyan Breezeway

POSTER SESSION B

B-1 Lemanik, Stephanie, Ontario Ministry of the Environment
Development of a Pesticides Screening Method Using Gas Chromatography/Time-of-Flight Mass Spectrometry

B-2 Zintek, Lawrence, U.S. EPA**,**
Standard Test Method for Determination of Nonylphenol, p-*tert*-Octylphenol, Nonylphenol Monoethoxylate and Nonylphenol Diethoxylate in Environmental Waters by Ultra Performance Liquid Chromatography/Tandem Mass Spectrometry

B-3 Antonious, G.F., Kentucky State University
Gas Chromatographic/ Mass Spectrometric Analysis of Capsaicin

B-4 Lim, Moo Song, Gyeongin Regional Korea Food and Drug Administration
Monitoring of Neonicotinoid Pesticide Residues in Agricultural Products by LC/MS

B-5 Lee, Hee Jung, Busan Regional Korea Food & Drug Administration
Monitoring of EBI Pesticide Residues in Agricultural Products by GC/MS/MS

B-6 Lee, Jin-Ha, Daejeon Regional Korea Food & Drug Administration
Monitoring of Pesticide Residues in Special Products of Korea

B-7 Phillips, Tom, Maryland Department of Agriculture
Determination of Rodenticides in Area Wipe Sample by LC-UV/Fluorescence LC-MS/MS

B-8 Bauder, Cathy, Schaner, Angela, Steward, Katie, Montana Department of Agriculture
Metabolites in Vegetation Using Solid Phase Extraction and Liquid Chromatography/Mass Spectrometry/Mass Spectrometry

B-9 Verreth, Jona, Schaner, Angela, Steward, Katie, Montana Department of Agriculture
Determination of Fluroxypyr, 3,5-Dichloropyridinol and Methoxypyridine in Soil by Solid-Phase Extraction and Liquid Chromatography-Electrospray/Mass Spectrometry/Mass Spectrometry

B-10 Schaner, Angela, Steward, Katie, Montana Department of Agriculture
Determination of Glyphosate and AMPA in Vegetation by LC/MS/MS

B-11 Schaner, Angela, Steward, Katie, Montana Department of Agriculture
Determination of Glyphosate and AMPA in Soil by LC/MS/MS

B-12 Allum, Raymond, Florida Department of Agriculture and Consumer Services
GC/TOF Analysis of Pesticides in Fruits and Vegetables

B-13 Iijima, Kazauki, The Institute of Environmental Toxicology, Japan
A Novel Matrix-Matching Method by Sequential Injection Technique with PTV-GC/MS

B-14 Thurman, Earl Michael, University of Colorado
Analysis of Phytoestrogens in Food and Wastewater by GC/MS

B-15 Yeung, Po Ying, Mathis, John A, Global Laboratory Services
Determination of pentachlorophenol and trichlorophenol using QuEChERS and SPE

B-16 Audrey Chen, FMC Corporation
Lesson Learned – Developing Residue Analytical Methods for Triazole (Conazole) Pesticides in Crop Matrices

ABSTRACTS

(A=POSTER SESSION A, B=POSTER SESSION B, C=PROGRAM COMMITTEE, M=MODERATOR)

ORAL PRESENTATIONS

01,

Yang, Paul, Ontario Ministry of the Environment, 125 Resources Road, Etobicoke, , Canada ON,
paul.yang@ontario.ca

Matrix Effects in Multiresidue Liquid Chromatography/Electrospray Ionization Tandem Mass Spectrometric Analysis of Emerging Organic Pollutants

The study on emerging organic pollutants (EOPs) in the environment has drawn much attention since the turn of the century. Liquid chromatography/electrospray ionization tandem mass spectrometry (LC/ESI-MS-MS) technique is demonstrated to be a powerful tool for the determination of these EOPs. However, due to the complex nature of environmental samples, matrix effects are a common issue in the analysis that is impossible to completely eliminate. In this presentation, we explored matrix effects during LC/ESI-MS-MS analysis of EOPs in detail and demonstrated the best approach to compensate for these effects. Our experimental results showed matrix effects existed in the form of suppression or enhancement of the electrospray ionization efficiency. The level of suppression or enhancement of certain EOPs would be affected by sample type, volume, pH, sample extract storage time, and the ionization mode employed. The use of isotope-labelled analogues provided the best tool to offset matrix effects for native EOPs. With more and more isotope-labelled analogues available, the new labor and time-saving strategy is to use isotope-labelled surrogates to correct for matrix effects and obtain accurate, high-quality analytical data.

02,

Chinchilla, Christina, Masis, Mario, Universidad De Costa Rica, , , , Costa Rica ,
cristina.chinchilla@ucr.ac.cr

Implementing ISO 17025 Accreditation in Costa Rica Laboratories

Since its creation more than twenty years ago the Centro de Investigación en Contaminación Ambiental (CICA) of the Universidad de Costa Rica has been focused in the development and adaptation of methods and novel analytical techniques to the Latin-American conditions and in training activities related to the analysis of trace pollutants. In 1995 CICA's laboratories initiated the implementation of a quality vision and with the support of the International Atomic Energy Agency the first Quality assurance-Quality control (QA/QC) system was established, nowadays the system has evolved through several steps to ISO 17025:2005 and the accreditation in more than 50 assays had been obtained under this norm. The first accreditation was obtained under the Guide ISO 25 in 2000 and CICA's Pesticides Residue Lab was the first to obtain this accreditation in the country.

The accreditation in the Pesticides Residue Analysis Lab includes assays for the determination of residues of more than 100 pesticides families in more than 30 commodities, water, soil, sediments and organisms. Since 2000 several technicians and scientists from all Latin America have been trained at CICA in pesticides residue analysis and QA/QC issues. The complexity of the analysis and the hard work associated to the implementation of QA/QC elements on daily routine work has lead to a process of constant training and continual improvement that will be discussed in the presentation.

03,

Jenkins, Ron, USDA-GIPSA, 10383 N. Ambassador Dr., Kansas City, MO, USA 64154,
g.ronald.jenkins@usda.gov

Two Case Studies: Validating Methods to Detect Inadvertently Released Grains into the Marketplace

The production of biotechnology-derived foods has increased in the United States over the past several years. Foreign countries that import US grains have not approved some biotechnology-derived traits that are approved for human consumption in the US. Indeed, governmental agencies abroad have passed legislation requiring labeling when biotechnology-derived food products that get introduced into their marketplaces exceeds a specified threshold level. In response to industry concerns, the Grain Inspection, Packers and Stockyards Administration (GIPSA) is taking an active role in standardizing procedures for detecting biotechnology-derived traits in grains and oil seeds. To manage risks, the industry needs testing methods that are accurate, reliable, cost effective and market compatible. Method Validation is an essential component and one of the measures universally recognized as a necessary part of a comprehensive system of quality assurance in analytical chemistry and food biotechnology. The inadvertent release of two unapproved traits, Bt-10 in maize and LL601 in rice, consequently disrupted grain trade abroad. The biotechnology workgroup at USDA-GIPSA played a major role in verifying accurate and reliable testing methods for the detection of trace amounts of these two traits. This presentation reflects on various aspects of method validation that need to be considered when polymerase chain reaction (PCR) is employed to detect and quantify the presence of biotechnology-derived traits in grains and oilseeds.

04, 09,

Mastovska, Katerina, USDA-ARS-ERRC, 600 E. Mermaid Lane, Wyndmoor, PA, USA 19038,
katerina.mastovska@ars.usda.gov

Pesticide multiresidue analysis in cereal grains using modified QuEChERS method

The QuEChERS sample preparation method was optimized and validated for the analysis of almost 200 pesticides in cereal grains (wheat, rice, oat, and corn) and other matrices. The pesticides were analyzed by two state-of-the-art GC- and LC-MS methods. The GC-MS employed large volume injection using automated direct sample introduction (DSI) technique combined with full-spectra data acquisition with time-of-flight (TOF) MS. In LC-MS, ultra-performance LC (UPLC) technique was used in combination with tandem MS for fast and selective analysis of LC-amenable pesticides. The described analytical approach is currently being used for routine multiresidue analysis of pesticides in various food matrices.

05,

Simonds, Roger, USDA-AMS-S&T-National Science Lab, 801 Summit Crossing Place B, Gastonia, NC, USA 28054, *roger.simonds@usda.gov*

Pesiticide Residue Analysis of Honey Bee Products Using Quechers

The USDA-AMS-Science & Technology Program-National Science Laboratory in Gastonia, NC has been analyzing honey bee products for pesticide residues in support of research into Colony Collapse Disorder (CCD) since early 2007. The types of honey bee products being analyzed are as follows: honey, pollen, beeswax, royal jelly, bees, brood, wax foundation, and bee bread. Slight modifications of the Quechers method were utilized for extracting pesticide residues from these products. The analyses were performed using LC/MS/MS, GC/MS-EI, and GC/MS-NCI (Negative Chemical Ionization). The results of the analyses are discussed as well as the nuances that each product presents and how they are addressed with modifications to the Quechers method. GC/MS-NCI is discussed in detail as an analytical tool that is very valuable for analyzing many pesticide residues at low levels in complex matrices.

06, C,

Wong, Jon, US Food and Drug Administration, 5100 Paint Branch Parkway HFS-706, College Park, MD, USA 20740, *jon.wong@fda.hhs.gov*

A modified QuEChERS procedure for the uPLC-MS/MS analysis of pesticides in fresh produce

A method employing QuEChERS and determination using ultraperformance liquid chromatography- tandem mass spectrometry (uPLC-MS/MS) was developed for the multiresidue analysis of pesticides in fruits and vegetables of high water and low fat content. The QuEChERS procedure was modified by incorporating graphitized carbon and primary secondary amine sorbents to remove matrix co-extractives. This method is also amenable for GC or GC-MS analysis. For the ~200 target pesticides studied, uPLC-MS/MS conditions were determined and most of the recoveries were between 70 - 120%, with relative standard deviations below 20% for fortification concentrations of 25, 100 and 500 µg/kg. The method was used to quantitate and identify various incurred residues such as carbamates, benzoylureas, triazoles, and/or macrocyclic lactones at levels ranging from <1 – 600 µg/kg in produce samples.

07, C,

Lehotay, Steven, USDA ARS, 600 East Mermaid Lane, Wyndmoor, PA, USA 19038, *steven.lehotay@ars.usda.gov*

Effect of Chlorophyll on Long-Term GC Performance in Pesticide Residue Analysis

A common perception among pesticide residue analysts is that chlorophyll co-extracts cause problems in long-term GC performance. We have conducted experiments to compare and assess the chromatographic quality for multiple pesticides in spinach QuEChERS extracts injected by different techniques in GC-MS (SIM). One approach is automated direct sample introduction (DSI), and the other is programmable temperature vaporization (PTV), both using large volume injection (LVI). One set of extracts was treated with graphitized carbon black to remove chlorophyll, and the other was not (both using dispersive-SPE with primary secondary amine sorbent). Multiple sequences were conducted to determine the effects on calibration standards in the spinach extracts.

08, C,

Schenck, Frank, FDA, 60 Eighth St NE, Atlanta, GA, USA 30309, *Frank.Schenck@fda.hhs.gov*

OP pesticides at ppb levels using QuEChERS & GC-PFPD

The vulnerability of infants and children to organophosphorus pesticides (OPs) warrants the use of analytical methods capable of detecting them in foods at trace levels. An MRM for the analysis of OPs in produce at levels down to 1.0 µg/kg (1.0 ppb) has been developed using a modification of the QuEChERS method. Pesticides were extracted with acetonitrile, salted-out. And cleaned-up using dispersive SPE with PSA & GCB sorbents. Analysis was by GC with pulsed flame photometric detection. Recoveries of 102 OPs, at 1.0, 10 and 100 ppb, in 4 commodities ranged from 62-125%, with >80% being achieved for most of the pesticides. Over 400 produce samples collected from a cohort of children that participated in the Children's Pesticide Exposure Study (CPES) and the Longitudinal Dietary Pesticide Exposure Study (LDPES) were analyzed. Residues of 15 of the102 pesticides tested were detected and quantified, at levels ranging from

10, 11,

Helen Stahnke, Alder, Lutz, Federal Insitute for Risk Assessment, Thielallee 88-92, Berlin, Germany D-14195, *lutz.alder@bfr.bund.de*

No more trouble with matrix effects? A simple way to consider matrix effects in ESI-MS without standard addition, matrix matched or stable isotope labelled standards

Matrix effects are well-known in electrospray ionization. A poor accuracy of quantitative data is obtained, if the ionization efficiency is changed compared to standards (in solvent). We studied matrix effects for a large number of pesticides in 20 food matrices of plant origin. At first the simplest "dilute and shoot" strategy was studied. In a next step the impact of identical matrices on different analytes was thoroughly studied using a post-column infusion system. We were able to compare matrix effects of pesticides with different retention times under identical conditions. We made a surprising observation, the electrospray ionization of all pesticides was influenced in the same manner by the diverse (hidden) matrix components. That means the seemingly "unpredictable behaviour" of matrix on the ESI response of diverse pesticides is mainly caused by the different chromatographic behaviour (different pesticides co-elute with different matrix components). Since profiles are very similar, it is not necessary to determine matrix effect profiles for each individual compound. The effect of matrix on any analyte can be successfully predicted by infusion of a single "monitor substance". Its profile allows (i) to detect regions in the chromatogram without matrix effects, (ii) to estimate, if "dilute and shoot" strategy requires low dilution only, and (iii) to predict the extent of matrix effects to decide upon the violation of MRLs.

12,

Dimandja, Jean-Marie, Spelman College, Atlanta, GA, *jdmandja@spelman.edu*

Comprehensive Two-Dimensional Gas Chromatography/Time-of-Flight Mass Spectrometry: Advances and Perspectives for Environmental and Bioanalytical Screening.

Comprehensive two-dimensional gas chromatography (GCxGC) is progressively emerging as a novel and exciting technology for the enhanced analysis of complex industrial, environmental, or bioanalytical samples. GCxGC methods are able to provide high-resolution separations in a time comparable to one-dimensional GC, while conserving or improving analyte detectability. In addition, compounds are also separated in terms of their chemical structure, which is very useful in many screening protocols. In this presentation we will describe the instrumentation and advantages of GCxGC when combined with time-of-flight mass spectrometry (TOF MS). An overview of GCxGC pesticide applications will also be presented, and the scope of GCxGC/TOF MS for a wider range of environmental toxicants will also be discussed.

13,

Ferrer, Imma, CEMS, University of Colorado, ECOT 441, 428 UCB, Boulder, CO, imferrer@ono.com , *imferrer@ono.com*

Multi-residue Methods using LC/TOF-MS for the Analysis of Pesticides in Food

Liquid chromatography coupled to mass spectrometry using time-of-flight (LC/TOF-MS) is an excellent technique for the analysis of pesticides in food. The high degree of confirmation by accurate mass measurements for target pesticides has demonstrated the applicability of the method in routine analysis, which is important in production laboratories. Moreover, the use of TOF-MS allows for non-target identification, because the full-spectrum is recorded at all times, which is not possible with MRM techniques. A comprehensive multi-residue method for the analysis of a large number of pesticides and their degradation products in fruits and vegetables is reported here. This technique was successful for finding non-targets and unknowns that have not been previously included in routine target methods. In conclusion, this talk presents an overview of the main achievements by LC/TOF-MS for food analysis including examples of identification of pesticides, additives and non-targets as well.

14,

Barr, Dana, CDC, 4770 Buford Hwy, Mailstop F17, Atlanta, GA, USA 30024, *dbarr@cdc.gov*

Biomonitoring of human exposure to pesticides

Pesticides have been used since the early to mid twentieth century. In the U.S. alone, over 600 pesticide active ingredients are formulated in about 21,000 different commercial products. Although many public health benefits have been realized by the use of pesticides, their potential impact on the environment and public health is substantial. Using three separate analytical methods, each requiring only 2 mL of urine, we can successfully measure 35 different pesticides or metabolites at background levels with a high degree of selectivity and precision. We describe a comprehensive approach to biological monitoring of current-use pesticides in urine using high-performance liquid chromatography-tandem mass spectrometry (HPLC-MS/MS) and gas chromatography-MS/MS with quantification using isotope dilution. In addition, we describe the unique complexities involved in interpreting biomonitoring data and how these data can be used for several public health and risk assessment applications.

15, 17, M,

Holmes, William, Mississippi State University, PO Box CR, MS State, MS, USA 39762, *wholmes@ra.msstate.edu*

Ethanol Fuel: The Impact of Aflatoxin Degradation

The ethanol fuel industry has become an important partner of American agriculture and increased public demand for ethanol as renewable fuel has triggered a tremendous increase in corn production. A. flavus is a fungus that can

infect corn, peanuts, cotton, and tree nuts; it produces the potent mycotoxin, aflatoxin. Aflatoxins are toxic and carcinogenic compounds regulated by the FDA. Strict exposure limits of 20 ppb for human consumption are enforced both in the US and abroad, resulting in millions of dollars of unusable crops annually. Aflatoxin contamination becomes increasingly problematic when corn is used for ethanol production. Although aflatoxin has not been reported in ethanol produced from contaminated products, the resulting distiller's grains show increased concentration of 3-4 times the initial level of aflatoxins. Thus, we have initiated studies evaluating degradation of aflatoxin.

16,

Kamel, Alaa, Environmental Protection Agency, 701 Mapes Road, Fort George G. Meade, MD, USA 20833, *kamel.alaa@epa.gov*

Development and validation of a multi-residue method for the analysis of neonicotinoid and macrocyclic lactone pesticides in milk and 11 fruit and vegetable commodities by LC-MS/MS

A multi-residue method has been developed and validated for the detection of thirteen neonicotinoid pesticides and metabolites, in addition to nine macrocyclic lactone pesticides and veterinary drugs using solid phase extraction and liquid chromatography-tandem mass spectrometry (LC-MS/MS). The method has been validated at the limit of quantitation (LOQ), 2x LOQ and 10x LOQ in milk, orange, spinach, apple, plum, watermelon, green bean, zucchini, broccoli, strawberry, grape and tomato. The results show good recovery of the analytes, except for some neonicotinoid metabolites. The limits of detection (LOD) for all analytes ranged between 0.1 – 5 ng/g. This method was developed to collect monitoring data to assess the risk from dietary exposure to these pesticides.

18,

Payá, Paula, Faculty of Chemistry, University of Murcia, Campus Espinardo, s/n Dpt. Agricultural Chemistry, Geology and Pedology, Espinardo, Murcia, , Spain 30100, *paulapp@um.es*

Dissipation studies of pesticide residues in fruits and vegetables and human risk assessment

Pesticide persistence in field is influenced by weather conditions, commodity type and pesticide class and these are of great importance in the dissipation of residues for raw commodity ingestion. However, some manipulation and manufacturing processes suppose positive effects on the residue dissipation. The data obtained from these studies allow the determination of the so-called "transfer factor" in processed foods. The presentation will show data on the field and processing dissipation of pesticides belonging to different classes (organochlorine, organophosphate, carbamates, insect growth regulators, etc). These studies were carried out on commodities such as broccoli, lettuce, pepper, tomato, apricot, peach, orange, mandarin, grape, barley, etc. Finally, the estimated daily intake will be reported to set comparison with the acceptable daily intake for some pesticides. In vitro bioavailability trials in foods will be exposed too.

19,

Brewer, William, University of South Carolina, Dept. of Chemistry and Biochemistry 631 Sumter Street, Columbia, SC, USA 29208, *brewer@mail.chem.sc.edu*

Multi-Residue Pesticide Analysis on Incurred Produce Samples using Variations of Disposable Pipette Extraction

Strategies for extracting multiple pesticides in fruit and vegetables have been studied. Using Disposable Pipette Extraction (DPX), various types of pesticides can be rapidly analyzed with high efficiency. The types of DPX products used in these studies include DPX-RP (reverse phase), DPX-Q (QuEChERS), and DPX-L/L (liquid-liquid solid phase extraction). The compounds that appear to give difficulty using DPX-Q (such as chlorothalonil) are readily extracted using DPX-RP, and very polar compounds that are difficult to analyze by DPX-RP are readily extracted using DPX-Q. Hence, the DPX methods are complimentary. Results of analyses using acetone and acetonitrile as initial solvents will be presented. Advantages and disadvantages of both solvent systems will be determined using GC/MS, GC-NPD and GC-ECD detectors. The results using DPX will be compared to the Modified Luke Method for the analysis of incurred samples. In addition, use of complete automation will be discussed using the GERSTEL MPS-2 instrumentation.

20,

André de Kok, National Reference Laboratory for Pesticides Analysis, Amsterdam, The Netherlands. *andre.de.kok@vwa.nl*

Comparison of HPLC- with UPLC-MS/MS
Multiresidue Methods for the Determination of
Pesticide Residues in Primary Agricultural Products:
Extension of Scope (>220 pesticides) vs. Shorter Run Times

As the first aim of a comprehensive research study, the scope of our LC-MS/MS multiresidue method [Multiple Reaction Monitoring (MRM)-mode] used in daily routine for the determination of 170 pesticides in 4500 samples yearly, has been extended by using the latest generation, fast scanning triple-quadrupole tandem mass spectrometer (Waters Quattro Premier). The ability to apply higher scan rates (lower dwell times) allows the inclusion of more than 220 pesticides in one 30-min. chromatographic run, with at least two MRM transitions per pesticide for the required confirmation (with permitted ion ratio tolerances). Alternatively, by sacrifying only a limited number of transitions and

still maintaining a very wide scope, the combined scanning in the positive and negative electrospray ionisation mode is now practically achievable. The selection of MRM transitions (precursor-daughter ions) and MS/MS acquisition parameters (cone voltage, collision energy) were optimised by infusion of standard solutions into the tandem mass spectrometer. For all pesticides, the positive-mode and for a selected group, both positive- and negative-mode electrospray ionisation were tested.

21,

Zintek, Lawrence, US EPA, 536 South Clark Street, Chicago, IL, USA 60605, *zintek.lawrence@epa.gov*

Methods for Extractable Nonvolatile Compounds Analyzed by Liquid Chromatography/Mass Spectrometry for Environmental Restoration Following Homeland Security Events

The US EPA needs standardized methods for qualitative and quantitative determination of target analytes of concern during environmental restoration. This presentation will demonstrate four quick and robust liquid chromatography tandem mass spectrometry (LC/MS/MS) methods for detecting and quantifying a series of extractable nonvolatile compounds, including some degradation products of chemical warfare agents. These methods focus specifically on analyzing organophosphonic acids, carbamates, ethanolamines and thiodiglycols in water samples, targeting risk levels provided by NHSRC. EPA's National Homeland Security Research Center (NHSRC) is currently validating these four draft procedures in reagent and environmental water samples. The US EPA Region 5 laboratory is concurrently developing analytical methods for the detection of these analytes in soil and wipes.

22,

Podhorniak, Lynda, EPA/OPP/Analytical Chemistry Lab, 701 Mapes Road, Fort Meade, MD, USA 20755, *podhorniak.Lynda@epa.gov*

Recent Analytical Challenges: Proportionality Studies

As a requirement of the pesticide registration process, crop field trials are conducted to determine the level of pesticide residue found on crops at the maximum labeled application rate (1X) of a pesticide. The residue data are then used by the EPA to establish a tolerance for the pesticide. A study was initiated to determine whether, in fact, a proportional relationship exists between application rate and resulting pesticide residues. The study was designed using side-by-side field trials of various crops treated at 1X, ½ X, 2X and 4X the labeled application rate. Six different pesticides were selected for this study and six different single-analyte methods were used to quantitate the residues. Problems were encountered with several of the methods as written and modifications of those methods needed to be made prior to analyzing the samples for the study. This presentation will focus on the analytical challenges encountered and how we modified the methods to get acceptable results.

POSTER SESSION A

A-01,

Schreiber, Andre, Applied Biosystems / MDS Sciex, 71 Four Valley Dr, Concord, Ontario, , Canada L4K4V8, *andre.schreiber@appliedbiosystems.com*

LC/MS/MS for the Quantitation and Confirmation of Endocrine Disrupting Compounds, Pharmaceuticals and Personal Care Products in Water Samples with Library Search Confirmation

Nadia Pace, Christopher Borton, Hesham Ghobarah, Jim Krol

This presentation highlights recent progress in developing a screening method for the detection of EDC and PPCP in waterways. Special emphasis is on confirmation of identified and quantified contaminants in water samples using MRM ratio and mass spectral library searching. The method in this work was developed on a hybrid triple quadrupole linear ion trap mass spectrometer (4000 QTRAP LC/MS/MS system) fitted with an ESI source in positive and negative polarity. All compounds were detected in Multiple Reaction Monitoring (MRM) mode and Information Dependent Acquisition (IDA) experiments were used to trigger Enhanced Product Ion (EPI) spectra. These spectra were searched automatically against a mass spectral library of EDC and PPCP. The mass spectral library was generated by injection of standards of investigated compounds using a standardized Collision Energies (CE) of 35V with a Collision Energy Spread (CES) of 15V. Water samples collected in upstream and downstream of water treatment plants were collected and concentrated using Solid Phase Extraction (SPE) and then analyzed by LC/MS/MS. The standardized collision energies used to produce the library were also used to acquire the analytical data of water samples. Up to a hundred endocrine disrupting compounds, pharmaceuticals, and personal care products were quantified and confirmed by library searching. Detection and quantitation of all compounds was achieved down to low part per trillion levels (ng/L). Identified and confirmed contaminants are compared in both upstream and downstream samples to evaluate the effectiveness of the water treatment for PPCP and EDC.

A-02,

Schreiber, Andre, Yuriko Ozeki, CJ Baker, Applied Biosystems / MDS Sciex, 71 Four Valley Dr, Concord, Ontario, , Canada L4K4V8, *andre.schreiber@appliedbiosystems.com*

Using the MRM catalogue of Cliquid software to quickly build custom LC/MS/MS methods for pesticide analysis matching the new Japanese Positive List

Recent regulations on food and environmental analysis especially in Europe and Asia require the screening for pesticides using GC/MS and LC/MS/MS. With more than 1000 pesticides and metabolites being in use or present in the environment there is a demand for powerful and rapid analytical methods, which can detect low concentrations.

Presently, no analytical technique is able to detect so many analytes in a single method. Here the new Cliquid software provides an easy way of developing such screening methods. Built into the software is an MRM catalogue containing presently more than 500 compounds which can be used to quickly build LC/MS/MS method based on MRM transitions, compound dependant parameters and retention time information saved into the catalogue. The presentation shows an example of rapid method development of 4 pesticide screening methods matching new Japanese food testing regulations for the analysis of more than 200 pesticides in various food matrices.

A-03,

Schreiber, Andre, Doina Caraiman, Nadia Pace, CJ Baker, Applied Biosystems / MDS Sciex, 71 Four Valley Dr, Concord, Ontario, Canada L4K4V8, *andre.schreiber@appliedbiosystems.com*

Accelerated LC/MS/MS for the Quantitation and Confirmation of Pesticides in Food and Water Samples

Recent regulations on food and environmental analysis require the screening for pesticides using confirmatory techniques, such as GC/MS and LC/MS/MS. With more than 1000 pesticides of more than 100 compound classes and their metabolites and degradation products being in use or present in the environment there is a demand for powerful and rapid analytical methods, which can detect very low concentrations of pesticides. Here we present a high-throughput LC/MS/MS method that combines the high sensitivity MRM screening with fast EPI confirmation and allows detection of low concentration pesticides, with LOD below 0.1µg/L and direct injection of water samples. The developed LC/MS/MS method was used to monitor pesticides in various fruit and vegetable samples after simple QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) extraction1-3. Additionally, water samples were injected directly into LC/MS/MS and pesticides could be detected at concentration levels of 0.1µg/L or below. In one single experiment up to 600 MRM transitions were monitored without cross talk using traditional MRM and scheduled MRM (sMRM) functionality.

A-04,

Hubbard, Nik, Varian, Inc., 13000 Weston Parkway, Cary, NC, USA 27513, *nik.hubbard@varianinc.com*

Multi-Residue Analysis of Pesticides in Vegetable and Fruit Extracts by Gas Chromatography Mass Spectrometry

A general maximum residue level of 0.01mg/kg for pesticide residues is proposed in the current EU member State legislation on pesticide Maximum Residue Levels (MRLs) in food and feed of plant and animal origin (1). Analytical laboratories prefer sample preparation without elaborate cleanup steps. Excellent full scan can be achieved in this demanding matrix while maintaining instrument robustness. An EI full scan GC/MS method was developed for the determination of 80 pesticides in a mixture of six vegetable and fruit matrices. The linearity of pesticides was studied over the concentration ranges of 5, 10, 20, 50, 100, 200 and 500 ppb with internal standard at 100 ppb in the mixed vegetable and fruit matrices. This range covers current EU member State legislation on pesticide Maximum Residue Levels (MRLs). Results confirmed that the method developed can determine most of pesticides quantitatively as low as 5 ppb with excellent % RSD's.

A-05,

Beck, Jonathan, Yamagishi Y, Japan, Schoutsen FWP, The Netherlands, Thermo Fisher Scientific, 525 W Aldine Ave 401, Chicago, IL, USA 60657, *jonathan.beck@thermofisher.com*

Increased Mass Resolution to Reduce Matrix Interference with the LC-MS/MS Analysis of Pesticides in Food Residues

There is an increased concern about food safety and the number of regulated pesticide residues has risen. Thus there is a need for an accurate, high sensitivity and high throughput screening method which can quantitate a large number of pesticide residues. Because of the identification and quantitation capabilities the LC-MS/MS, it is becoming the technique of choice. However the different and complex matrices in these analysis can make it very challenging to quantitate pesticide residues. Although the sample clean up procedures can remove a lot of matrix related interferences, the analytical instrument has to be highly selective and sensitive. Increased resolution has proven to be very useful for this purpose. The analytical method shows an analysis of 35 pesticide residues in food matrices.

A-06,

Phillips, Eric, Thermo Fisher Scientific, 2215 Grand Ave Pkwy, Austin, TX, USA 78728, *eric.phillips@thermofisher.com*

Multi-residue Pesticide Analysis Using Ion Trap GC/MS/MS Analysis with a Modified QuEChERS Extraction for Lettuce

The determination of pesticides in fruits and vegetables has been simplified by QuEChERS sample preparation method, published recently as AOAC Method 2007.01. The sample preparation is shortened by using a single step buffered acetonitrile (MeCN) extraction and liquid-liquid partitioning from water in the sample by salting out with sodium acetate and magnesium sulfate (MgSO4). The extract is solvent exchanged to hexane/acetone for splitless injection with detection by EI MS/MS. The system was qualified using endrin as a test compound. The study was performed to determine the linear range and detection limits for a list of pesticides, commonly used on iceberg lettuce crops, prepared in matrix using the QuEChERS sample preparation guidelines. A splitless injection was made with detection in EI MS/MS. The calibration curves for the pesticides studied met a linear least squares calibration with a

correlation coefficient of R2 > 0.99 for most compounds. The Method Validation Study generated an average % RSD of 9.7% for four replicate analyses at 50 ng/g and a calculated average LOD of 2.7 ng/g in iceberg lettuce based on 8 replicate analyses of 5 ng/g.

A-07,

Beck, Jonathan, Thermo Fisher Scientific, 525 W Aldine Ave 401, Chicago, IL, USA 60657, *jonathan.beck@thermofisher.com*

Comparing Large Injection Volumes and Online Pre-concentration in Water Analysis

A comparison for online sample preconcentration of 1mL, 5mL and 20mL injections of drinking water samples spiked with pesticide compounds is demonstrated. Two C18 LC columns are utilized, one acting as a loading or trapping column, the second as an analytical column for analysis of the compounds in the mixture. A six port valve is used to control whether the instrument is in a loading condition, or in an eluting condition. For separation prior to analysis using a LC-MS/MS instrument, HPLC is used. This method uses drinking water for direct injection onto the loading column, with no sample preparation or offline concentration.

A-08,

Stevens, Joan, Gilson, Inc., 3000 Parmenter Street, Middleton, WI, USA 53562, *jstevens@gilson.com*

Determination of Veterinary Drug Residues in Fish by an Automated SPE-HPLC System

The FDA is responsible for ensuring the safety of seafood, however efficient monitoring of aquacultured fish for residues is very limited. Optimizing the preparation of the sample for analysis not only lowers the LOD but enhances the quality of the results by lowering RSDs. The system presented is a totally automated SPE with HPLC analysis capabilities. The system allows for unattended sample preparations and on-line analysis via HPLC without manual intervention. Method performance was evaluated over several days of replicated samples of controlled salmon, salmon fortified with a drug mixture, and salmon dosed with a representative from several drug classes (quinolones, floroquinolones, macrolides, malachite green, imidazoles, tetracyclines, penicillin and betalactams). The complete automated system allows for ease of use and transfer of the method between sites minimizing down time and time required for validation and calibration

A-09,

Crawford, Mark, Gilson, Inc., 3000 Parmenter St, Middleton, WI, USA 53562, *mcrawford@gilson.com*

The Examination and Automation of GPC, SPE and QuEChERS for Pesticides in Oil

Several techniques for the separation of pesticides from edible oils are used today to investigate the amount of pesticides in these oils. GPC, SPE and QuEChERS can be used to isolate the pesticides that are found in edible oils. Each technique provides strengths for its separation of pesticides. GPC has the ability to process large amounts of sample, SPE provides disposable cartridges with numerous sorbents to provide separation of the analyte from the matrix, and QuEChERS involves uncomplicated sample cleanup of pesticides in aqueous matrices. This application investigates each of these separation techniques in separating pesticides from oil matrices and presents detailed information on the automation of each separation system.

A-10,

Wylie, Philip, Agilent Technologies, 2850 Centerville Rd., Wilmington, DE, USA 19808, *phil_wylie@agilent.com*

Evaluating a Method to Analyze Pesticides at the 10 ppb Level Using GC/MS with Large Volume Injection, Deconvolution Reporting Software, and a Retention Time Locked Pesticide Library

Fernandez-Alba A.R., Aguera A., Mezcua M., Martínez-Uroz M.A.
University of Almeria, 04071 Almeria, SPAIN

Most GC methods for pesticide residue analysis include a high temperature bake-out step to remove high boiling co-extracted matrix compounds, but this causes increased mass spec source contamination and shortens column life. With some dirty samples, chromatography can degrade after a few injections. This paper discusses a method for the analysis of pesticides using large volume injection with backflushing to remove high boiling materials from the head of the GC column. Backflushing with a QuickSwap shortens the run time, preserves the GC column, and keeps the MSD clean much longer. The scan data are analyzed using Deconvolution Reporting Software (DRS) which screens the results for 927 pesticides and endocrine disruptors. DRS can also be used to quantify compounds after deconvolution, which is especially useful for samples in heavy matrix. Pesticides found by DRS can be added to the list of target compounds to be quantified in future analyses. Most importantly, the method can be used to screen for very large numbers of pesticides and endocrine disruptors – not just a subset of targets. Approximately 50 – 75% of pesticides could be identified at or near 10 ppb depending on the matrix.

A-11,

Wylie, Philip, Agilent Technologies, 2850 Centerville Rd., Wilmington, DE, USA 19808, *phil_wylie@agilent.com*

Bridging the Performance Gap Between GC/MS and GC/MS/MS with Deconvolution Technology

Terry Sheehan, Melissa Churley, Paul Zavitsanos, Michael Szelewski

Although the selectivity of capillary GC/quadrupole MS has proven adequate for many types of analyses, the requirement for lower detection limits in increasingly complex samples has driven GC/MS to the limits of acceptable performance. NIST AMDIS (Automated Mass Spectral Deconvolution and Identification Software) is a mathematical solution to the selectivity limitation of GC/MS, but the degree to which deconvolution extends the boundaries of performance are not well documented. GC/MS/MS, the new standard for selectivity, has obvious performance advantages that are offset by the practical constraints of cost. The study goal is an assessment of the degree to which deconvolution software can bridge the gap between GC/MS and GC/MS/MS for trace analysis in complex separations with overlapping matrix peaks.

A-12,

Zweigenbaum, Jerry, Agilent Technologies, Inc., 2850 Centerville Road, Wilmington, DE, USA 19808,
J_Zweigenbaum@agilent.com

HILIC Separation of Herbicides and Other Quaternary Ammonium Compounds by LC/MS

Herbicides, such as paraquat and diquat, are used as defoliants and are highly toxic .Thus, their analysis at trace levels is of practical significance. Electrospray mass spectrometry (ESI-MS) of these and other quaternary ammonium herbicides such as difenzoquat provides a very strong signal. Separation is often difficult by conventional RP-HPLC but has been achieved with ion-pairing agents. However, even with volatile ion-pairing agents, the performance of LC-ESI-MS is impacted. Capillary electrophoresis has been successfully used but, because of the very low loadings with this technology, detection limits are high. Hydrophilic interaction chromatography (HILIC) should be an ideal coupling of LC and ESI-MS for polar and ionic compounds. However, because paraquat and diquat are doubly charged, very strong interaction is observed with the HILIC stationary phase. The separation and detection of these compounds will be described.

A-13,

Shofran, Brian, LECO Corporation, 3000 Lakeview Ave., St. Joseph, MI, USA 49085,
brian_shofran@leco.com

Rapid Analysis of Carbamate Pesticides using LC-TOFMS

Carbamate pesticides are widely used as insecticides or fungicides. The development of fast and reliable methods for the analysis of these pesticide residues is an important field of research. This demand has been stimulated by the continuous public concern over residues in the food supply and the environment. Traditionally the screening of unknown pesticides has been accomplished by GC-MS analysis. However, many of the newer classes of pesticides are polar, nonvolatile chemicals, which are not amenable to GC-MS analysis. The use of HPLC coupled to Time-of-flight mass spectrometry (TOFMS) has become more commonly used for the determination of these types of pesticides. TOFMS provides a way to measure the accurate masses of analytes. Applications in which high mass accuracy is desirable include monitoring of target analytes in complex mixtures and identification of unknowns. An LC-TOFMS method was established for the rapid separation and identification of carbamate pesticides.

A-14,

Hilton, Donald, LECO Corp, 14950 Technology Ct., Fort Myers, FL, USA 33912,
Don_Hilton@LECO.COM

Screening for Environmental Contaminants in Complex Matrices - Tobacco

Contamination of materials intended for consumer consumption may be difficult to determine in complex matrices. Tobacco is a particularly challenging example, as it contains plant metabolites from the tobacco leaf as well as degradation products resulting from aging of tobacco and processing. While target analytical techniques can reliably identify many compounds, the reliability depends on sufficient separation of components in the mixture for the particular analytical technique. Target analyses are limited, however to the target list of compounds. With adequate separation and capability to rapidly evaluate the separated compounds, screening is possible - but automation is required in complex systems such as tobacco. Mathematical filters provide a way to evaluate mass spectral data and identify compounds by features identifiable in the mass spectrum. This work shows the application of this technique to tobacco.

A-15,

Young, Michael, Waters Corporation, 41 Lakeview Ave., Bellingham, MA, USA 2019,
michael_s_young@waters.com

SPE Protocol For Rapid UPLC-MS Determination Of Acidic Pesticides

Dispersive SPE cleanup using PSA (primary/secondary amino bonded silica) has been demonstrated for GC/MS or LC/MS determination of neutral or basic pesticide residues. However, this procedure (commonly known as Quechers) is not appropriate for determination of acidic residues. This presentation will discuss a PSA cartridge SPE cleanup procedure suitable for determination of acidic and base/neutral pesticides in the acetonitrile extract of fruits and vegetables. Neutral and basic pesticides are collected in a pass-through mode in a manner analogous to dispersion SPE since these compounds are not retained. The acidic compounds are retained on the sorbent and are eluted with a suitable eluent. Thus, both the base/neutrals and the acids are available to the analyst in two separate cleaned-up fractions derived from a single aliquot of the original sample. Also, we will discuss UPLC technology

coupled with tandem mass-spectrometry for rapid chromatographic analysis of these samples for acidic pesticides with no need for derivatization.

A-16,

Watts, Vivian, Chromsys LLC, 7686 Richmond Highway 111, Alexandria, VA, USA 22306, *vwatts@chromsys.com*

Comparing SIM and MRM for pesticide analysis in a complex grain matrix

GCMS/MS is a highly effective way of analyzing samples with complex/dirty matrices. Sensitivity and accuracy are superior to conventional GCMS due to higher signal to noise ratios and less interference. This is the result of virtually eliminating matrix interferences when for example running in MRM and Product Ion Scan mode. The Chromsys Evolution is an upgrade for the Agilent 5973 and 5975 GCMS systems, enabling users to do MS/MS work. Grain samples which are routinely analyzed for pesticide residue, typically have particularly complex matrices for which SIM mode is not always sufficient. To demonstrate the effectiveness of the Evolution upgrade (in Multiple Reaction Monitoring (MRM) mode) for analyzing trace analytes in a dirty matrix, a Solin extract sample was chosen as this matrix is known to present considerable matrix and sensitivity problems when using the traditional Single Ion Monitoring (SIM) mode of GCMS analysis.

A-17,

Settle, Virgil, GERSTEL, 1510 Caton Center Drive Suite H, Baltimore, MD, USA 21227, *vasettle@gerstelus.com*

Automation of Sample Preparation Steps Using a Robotic Autosampler

A single robotic XYZ coordinate autosampler commonly used for sample introduction in GC or HPLC can be used to perform a wide variety of sample preparation techniques using a single instrument and controlling software. The new MAESTRO software allows the user to control an expanded list of sample preparation techniques such as derivatization, saponification, esterification, analytical weighings, filtration, and solid phase extraction. In addition to ease of use and intuitive windows-based programming, the software includes tools to automate and optimize parameters assuring efficient sequence creation and maximum sample throughput. The sampler can be configured as part of a GC or LC system or can be configured as a benchtop workstation.

A-18,

Henderson, Sheldon, Dionex, 1182 West 2400 South, Salt Lake City, Utah, USA 84119, *sheldon.henderson@dionex.com*

ASE and Productivity Gains in Environmental Analyses

Accelerated solvent extraction (ASE) is an innovative approach to liquid-solid extraction. It is accepted under Method 3545A for the extraction of conventional environmental toxins such as PCBs, dioxins, PAHs, diesel range organics and chlorinated pesticides. It has also been effectively used for pesticide residues in plant and animal tissues, and more recently, brominated flame retardants in all environmental matrices. This technique uses elevated temperatures and pressures to achieve analyte extractions from solid or semi-solid matrices in about 15 minutes and with small volumes of solvents. ASE is fully automated and can facilitate in-line clean-up of samples using resins and adsorbents to retain co-extractables. Discussion will include ASE techniques to improve productivity in a lab setting to maximize overall sample throughput and reduce sample handling to minimize sample preparation time for a variety of environmental analytes and matrices.

A-19,

George, Johnny, Horizon Technology, Inc., 45 Northwestern Drive, Salem, NH, USA 3079, *jgeorge@horizontechinc.com*

Automated Solid Phase Extractions of Organochlorine Pesticides from Water

A-20,

Cochran, Jack, Restek, 110 Benner Circle, Bellefonte, PA, USA 16823, *Jack.Cochran@restek.com*

A New Capillary Column for the Analysis of Pesticides by GC/MS

Due to this increasing demand on pesticide analysts, there is a need for a comprehensive method for analysis of a broad range and prodigious number of pesticides. The GC/MS is a logical instrument to use as it provides a high degree of specificity and yet is relatively inexpensive and easy to operate relative to LC/MS/MS, high resolution GC/MS, and GC/MS/MS. The rich level of information provided by a conventional GC/MS not only helps discern between various analytes, but also helps to distinguish between an analyte and the often complicated sample matrices inherent in environmental and food samples. Equally important in setting up an effective pesticide analysis is the choice of capillary column, which needs, not only, to be of the proper selectivity to separate compounds which share spectral appearance, but also needs to exhibit minimal bleed and needs to posses a high degree of inertness. In this poster, a new capillary column with a silarylene based polymer that meets these requirements will be used in a PE Clarus 500 GC/MS to analyze a comprehensive list of pesticide types at low concentrations, in an attempt to illustrate the effectiveness of this column for this type of analysis.

A-21,

Robert Wohleb, Ian Wohleb and Marc Elliott, ILE, Inc., 989 Milton Ave., Ste. 1D, Ferndale, CA 95536 rwohleb@ile-inc.com

Immobilized Liquid Extraction of Organic Residues from Aqueous Matrices as a "Low Solvent" Sample Preparation Technique for GC/LC Analysis.

In this study, we investigated the use of Immobilized Liquid Extraction (ILE) for extracting organic compounds from aqueous environmental samples. ILE is a form of liquid/liquid extraction in which the "organic" phase is immobilized on the surface of an ILE device. As little as 100-300 uL of solvent are used per extraction. We evaluated extraction efficiency and detection limits for pesticides, TPH, PCBs and semivolatiles. We present data on the effect of complex matrix interferants, and evaluate increased throughput, decreased costs, greater precision, and reduced solvent usage and subsequent disposal.

A-22

Laura Chambers, Mike Duffy,OI Analytical, lchambers@oico.com

Using the Pulsed Flame Photometric Detector (PFPD) for Low-level Analysis of Organophosphorus Pesticides

Organophosphorus (OP) pesticides are among some of the most widely applied agricultural pesticides in the world, and as a result these toxic pesticide residues are present in many agricultural products, often at very low concentrations. This poster will illustrate the steps necessary to develop and validate an analytical method for OP pesticides using the PFPD. Calibration curves for some common OP pesticides will be shown, plus the expected linear range, minimum detectable amount, and calculation of statistical method detection limits (MDLs). Considerations for configuring the PFPD for phosphorus selective detection will be discussed, including using dual gate subtraction to eliminate any residual interference, and modified gate settings to extend the calibration range. PFPDView is used as a post-acquisition data processing tool.

POSTER SESSION B

B-01,

Lemanik, Stephanie, Ontario Ministry of the Environment, 125 Resources Road, Etobicoke, M9P 3V6, , Canada ON, *stephanie.lemanik@ontario.ca*

Development of a Pesticides Screening Method Using Gas Chromatography/Time-of-Flight Mass Spectrometry

Pesticides and their environmental degradation products are emerging issues that have drawn much attention since the last decade. Gas chromatography/time-of-flight-mass spectrometry (GC/TOF-MS) based instrumentation is a powerful technique that can be used for the screening of either known or unknown pesticides in a sample. We will present a GC/TOF-MS method that is capable of determining target pesticides and their degradation products in environmental samples. More than 200 pesticides, including organochlorines, triazines, organophosphates, and their degradation products were identified to demonstrate the capability of this method. Future method development will be discussed in terms of data quality and operational efficiency.

B-02,

Zintek, Lawrence, , U.S. EPA Region 5 Chicago Regional Laboratory, , Chicago, IL, USA , *zintek.lawrence@epa.gov*

Standard Test Method for Determination of Nonylphenol, p-tert-Octylphenol, Nonylphenol Monoethoxylate and Nonylphenol Diethoxylate in Environmental Waters by Ultra Performance Liquid Chromatography/Tandem Mass Spectrometry

Nonylphenol is an organic chemical produced in large quantities in the United States. It is soluble in water and moderately resistant to natural degradation in water. Because of its chemical properties and widespread use as a chemical intermediate, concerns have been raised over the risks it poses to both freshwater and saltwater organisms. ASTM Standard D7065-06, which is a liquid/liquid solvent extraction GC/MS method, meets the freshwater ambient water quality criteria of the Clean Water Act. A new proposed standard, ASTM D19-WK15211, is a solid phase extraction LC/MS/MS method which reduces the amount of sample required, solvents, and analysis time while meeting the saltwater ambient water quality criteria of the Clean Water Act. This new standard was tested on sea water, river water, secondary POTW effluent, and ASTM substitute wastewater. The resulting reporting limit was 100 ppt for nonylphenol, nonylphenol monoethoxylate, nonylphenol diethoxylate and p-tert-octylphenol.

B-03,

Antonious, George, Kentucky State University, 400 East Main Street 218 Atwood Research Center, Frankfort, KY, USA 40601, *george.antonious@kysu.edu*

Gas Chromatocraphic/ Mass Spectrometric Analysis of Capsaicin

Capsaicin [N-vanillyl-8-methyl-6-noneamide] is the most pungent of the group of compounds called capsaicinoids in chili peppers. Ninety Capsicum accessions selected from the USDA Capsicum germplasm collection were screened

for their capsaicinoids content using gas chromatography (GC/NPD). Fresh fruits were extracted with methanol and analyzed for capsaicin, dihydrocapsaicin and nordihydrocapsaicin. Mass spectrometric analysis of the fruit crude extracts indicated that the molecular ions at m/z 305, 307, and 293 which correspond to capsaicin, dihydrocapsaicin, and nordihydrocapsaicin, respectively, have a common benzyl cation fragment at m/z 137 that can be used for monitoring capsaicinoids in hot pepper fruit extracts. Concentrations of total capsaicinoids varied from not detectable to 11.2 mg fruit-1. Quantification of capsaicinoids allowed us to identify genotypes with high levels of total compounds for the mass production of capsaicinoids.

B-04,

Lim, Moo Song, Gyeongin Regional Korea FDA, 120, Juan-1dong, Nam-gu, Incheon, , Republic of Korea 402-835, *moosong@kfda.go.kr*

Monitoring of Neonicotinoid Pesticide Residues in Agricultural Products by LC/MS

Development of multi-residue analytical methods and monitoring for residual 6 neonicotinoid (acetamiprid, clothianidin, dinotefuran, imidacloprid, thiacloprid and thiamethoxam) pesticides in agricultural products were conducted. The methods for the pesticides analysis were developed using a LC/MS/MS. Residual levels of those pesticides were investigated in 851 agricultural products from 14 provinces in Korea. The linearity of the analysis was 0.9987-0.9998, and that of recoveries were 82-122%. According to the monitoring of the neonicotinoid pesticides in agricultural products, acetamiprid, imidacloprid, thiacloprid and thiamethoxam showed various levels (frequency of 327 detections) without excess of MRLs for Pesticides in Foods by the Korea Food Code. Further and continuous monitoring of the residual pesticides in agricultural products would be required. Therefore, this study could establish a rapid and effective monitoring way to investigate residual neonicotinoid pesticides levels.

B-05,

Lee, Hee Jung, Busan Regional Korea Food &Drug Administration, 123-7 Yongdangdong Namgu, Busan, , Republic of Korea (South Korea) 608-829, *sesamistreet@naver.com*

Monitoring of EBI Pesticide Residues in Agricultural Products by GC/MS/MS

To monitor pesticide residues of agricultural products circulating in Korea market, we analyzed samples. The monitoring of 8 pesticides by gas chromatography coupled to tandem mass spectrometry(GC/MS/MS) has been investigated. All of the samples were collected in different 7 cities (Changwon, Busan, Daegu, Gumi, Seoul, Ulsan, Pohang) from March to May 2008. The average recoveries obtained from each sample ranged between 86 and 129% at approximately 0.1 ppm concentration. As a result of monitoring the EBI pesticides in agricultural products, all pesticides (difenoconazole, diniconazole, fenarimol, fenbuconazole, hexaconazole, myclobutanil, nuarimol and paclobutrazol) were detected with various levels(frequency of 136 detections). But the results showed that the concentrations of detected pesticide residues for all samples were much lower than the maximum residue limits(MRL) established by Korean legislation.

B-06,

Lee, Jin-Ha, Daejeon Regional Korea Food & Drug Administration, Testing & Analysis Division, #120 Seonsa-ro, Seo-Gu, Daejeon, , Korea 302-828, *leejin@kfda.go.kr*

Monitoring of Pesticide Residues in Special Products of Korea

The experiment was carried out to analyze for their pesticide residues in 17 different types of the special products of geographical indication. We purchased 3 cereal grains, nuts and seeds, 3 fruits, 8 vegetables, mushrooms and other plants (Korean medicines) mainly at the agricultural cooperative's joints markets. Total 209 pesticides including multi-analyzed pecticides (204) and single-analyzed pesticides (5: Methamidophos, Acephate, Omethoate, Monocrotophos, Vamidothion) were analyzed by GC/MS/MS, HPLC/UVD (PDA) and GC/FPD. No. 83 method and single-analyzed method (Screening of multi-pesticide residue in the special products of geographical indication) of Korea Food Code was selected for validation in recovery and interferences of matrices, etc. The results were as follows: among the selected 17 special products, the residual pesticides were detected in 8 types of special products (40 in 302 samples, detection ratio: 13.2%).

B-07,

Phillips, Tom, Maryland Department of Agriculture, 50 Harry S. Truman Parkway State Chemist Section, Annapolis, MD, USA 21045, *phillitd@mda.state.md.us*

Determination of Rodenticides in Area Wipe Sample by LC-UV/Fluorescence LC-MS/MS

A gradient liquid chromatographic (LC) method for the determination of eleven anticoagulant rodenticides in area wipe samples was developed. A series of wipe samples were analyzed over a period of six days. Samples were dissolved in methanol with sonication and injection into an LC system having an ODS analytical column with absorption at 280 nm and confirmed by fluorescence. LC-MS/MS was also used to quantitate and confirm bromadialone. Injections were made by autosampler. The calculations were based on a linear curve of external standards made in the extraction solvent. The study analyzed 10 spike levels, and a representative blank for each compound. The concentration range was from 0.300 µg/wipe to 1300 µg/wipe. The results were analyzed by using a spreadsheet that calculated the recoveries. Recoveries ranged from 75% to 105% of quantitation by fluorescence and UV data. The MS/MS recoveries from bromadialone were from 85% to 102%.

B-08,

Bauder, Cathy, MT Dept. of Agriculture, Laboratory Bureau, McCall Hall Room 10 Montana State University, Bozeman, MT, USA 59717, *cbauder@mt.gov*

Determination of Pinoxaden and Two Metabolites in Vegetation Using Solid Phase Extraction and Liquid Chromatography/Mass Spectrometry/Mass Spectrometry

Pinoxaden is an herbicide used for post-emergence control of annual grasses in wheat and barley. After application the parent ester, pinoxaden, is rapidly hydrolyzed (< 1 day) to the NOA-407854 metabolite which in turn undergoes hydroxylation to the SYN-505164 form. The method presented determines the NOA-407854 and SYN-505164 metabolites in plant tissue. A 5 g sample is refluxed with 90/10 1 N HCl//ACN and a 0.5 mL aliquot subjected to clean up with an ENVI-carb solid-phase extraction cartridge. Determinations are by reverse phase liquid chromatography electrospray ionization and tandem mass spectrometry in the positive ion mode. Two precursor-product ion transitions are used to quantify and confirm results. Average recoveries ranged from 55-91% for NOA-407854 and 49-91% for SYN-505164.

B-09,

Verreth, Jona, MT Dept. of Agriculture, Laboratory Bureau, McCall Hall Room 10 Montana State University, Bozeman, MT, USA 59717, *,jverreth@mt.gov*

Determination of Fluroxypyr, 3,5-Dichloropyridinol and Methoxypyridine in Soil by Solid-Phase Extraction and Liquid Chromatography-Electrospray/Mass Spectrometry/Mass Spectrometry

ANGELA M. SCHANER

Fluroxypyr is a member of the pyridine class of herbicides and is an extremely effective herbicide used to control broadleaf weeds. The method presented determines fluroxypyr, 3,5-dichloropyridinol and methoxypyridine in soil. A 10 g sample is extracted with 50 mL 90/10 acetone/0.1N HCl and a 5 mL aliquot subjected to clean up on an ENVI-Carb solid-phase extraction cartridge. Determinations are by reverse phase liquid chromatography with both positive and negative electrospray ionization and tandem mass spectrometry. Two precursor-product ion transitions are used to quantify and confirm results. Average recoveries for soils tested ranged from 68-89% for fluroxypyr and 3,5-dichloropyridinol. Recovery for methoxypyridine was low and ranged from 28-59%

B-10,

Schaner, Angela, MT Dept. of Agriculture, Laboratory Bureau, McCall Hall Room 10 Montana State University, Bozeman, MT, USA 59717, *aschaner@mt.gov*

Determination of Glyphosate and AMPA in Vegetation by LC/MS/MS

Glyphosate is a broad spectrum, nonselective systemic herbicide used for the control of broad leaf weeds and grasses. It is frequently suspected in pesticide misuse cases due to its popularity and potentially damaging effects. This method utilizes isotopically labeled surrogate standards to negate matrix effects and employs reverse phase liquid chromatography electrospray ionization and tandem mass spectrometry (LC-ES/MS/MS) in the negative ion mode. A 10 g sample is extracted with water and a 0.475 mL aliquot derivatized with FMOC prior to clean up on an Oasis HLB solid-phase extraction cartridge. Two precursor-product ion transitions are used to quantify and confirm results. Average recoveries for plant tissue tested ranged from 59-86% with one exception: low AMPA (25-36%) recoveries for lilac leaves matrix.

B-11,

Schaner, Angela, MT Dept. of Agriculture, Laboratory Bureau, McCall Hall Room 10 Montana State University, Bozeman, MT, USA 59717, *aschaner@mt.gov*

Determination of Glyphosate and AMPA in Soil by LC/MS/MS.

Glyphosate is a broad spectrum, nonselective systemic herbicide used for the control of broad leaf weeds and grasses. Due to it's popularity and potentially damaging effects, it is frequently suspected in pesticide misuse cases. The method presented determines glyphosate and AMPA, the glyphosate degradate, using isotopically labeled surrogate standards. The method employs reverse phase liquid chromatography electrospray ionization and tandem mass spectrometry (LC-ES/MS/MS) in the negative ion mode. A 10 g sample is extracted with a phosphate ammonium buffer solution then subjected to clean-up with dispersive carbon. A 0.475 mL aliquot is derivatized with FMOC then taken through an Oasis HLB solid-phase extraction (SPE) cartridge for additional clean-up. Two precursor-product ion transitions are used to quantify and confirm results. Average recoveries for soils tested ranged from 62-99%.

B-12, C,

Allum, Raymond, FDACS, 3125 Conner Blvd Lab #3, Tallahassee, FL, USA 32399, *allumr@doacs.state.fl.us*

GC/TOF Analysis of Pesticides in Fruits and Vegetables

There is a big push for larger screens of pesticides in fruits and vegetables. Analyzing hundreds of compounds in a sample quickly and efficiently is a goal that is possible today. Utilizing Time of Flight Mass Spectrometry can accomplish this goal. The power of full scan analysis coupled with deconvolution has made it possible for a single

injection on the instrument that can identify many compounds. The experiment illustrates that TOF can attain the same levels of detection as a GC/MSD in SIM mode.

B-13,

Iijima, Kazauki, The Institute of Environmental Toxicology, 4321 Uchimoriya-Machi Mitsukaido, Ibaraki, , Japan 303-0043, *iijima@iet.or.jp*

A New Matrix-Matching Method by Sequential Injection Technique with PTV-GC/MS

We reported the problem of matrix effects in the multiresidue analysis by GC/MS at the FPRW-2007. The matrix effect resulting in the enhancement recoveries was more apparent at a low fortification level (0.01 mg/kg) in comparison with a high level (0.1 mg/kg) for most of the investigated pesticides. So the purpose of this study is to improve the quantitative accuracy at the low residue level by the matrix-matching method and a PTV injection technique. A new sequential injection technique developed for preparation of matrix-marched standards by a PTV injector was quick, simple and convenient to use. The matrix-matched standard solutions were automatically prepared by sequentially injecting aliquots of blank samples (1 µL) and solvent standard solutions (1 µL). Then both solutions are automatically mixed in the bottom of injection liner at 66□C (below solvent bp). The applicability of the method was evaluated for 90 pesticides by the recovery test of 10 fortified animal and fishery products at 0.01 mg/kg. The majority of the investigated pesticides gave excellent recoveries ranging from 70 to 120% as the median of mean recoveries using by the combination technique.

B-14,

Thurman, Earl Michael, University of Colorado, Dept of Civil, Environmental, Archtect. Eng. ECOT 441 428 UCB, Boulder, CO, USA 80309, *michael.thurman@colorado.edu*

Analysis of Phytoestrogens in Food and Wastewater by GC/MS

Plant phytoestrogens have been cited as possible endocrine disruptors in fish along with synthetic hormones and human estrogens from wastewater effluents. Because phytoestrogens are excreted, not only by plants but also by humans via food consumption, it is important to evaluate their contribution to wastewater and their role in endocrine disruption in fish. Thus, methods were developed by GC/MS with derivatization to evaluate 5 plant phytoestrogens in soy products and in wastewater from streams impacted by high soy-product use (i.e. Boulder Creek in Boulder, CO). The phytoestrogens (genistein, daidzein, biochanin A, formonotein, and coumestrol) are derivatized as their silyl ethers after isolation by liquid/liquid extraction using ethyl acetate. The compounds were analyzed both in food and water by GC/MS/MS using ion trap at m/z 486, 398, 428, 340, and 412 respectively for each of the five phytoestrogens. Results are reported from >1000 ug/L (soy milk) to 1 ug/L for wastewater.

B-15,

Yeung, Po Ying, Global Laboratory Services, 2107 Black Creek Rd, Wilson, NC, USA 27893, *yeungp01@globallaboratory.com*

Determination of pentachlorophenol and trichlorophenol using QuEChERS and SPE

Method development and optimization was performed for the determination of pentachlorophenol, trichlorophenol and other compounds in tobacco and related matrices. A rapid sample preparation method known as QuEChERS was used. The samples were diluted with water and processed with a solid-phase extraction procedure using a mixed mode weak anion exchange/reversed phase sorbent. A high performance liquid chromatography tandem mass spectrometry (HPLC-MS/MS) method with atmospheric pressure chemical ionization in the negative ion mode was used for analysis.

B-16, C,

Chen, Audrey, FMC Corp., , , , , *Audrey.Chen@fmc.com*

Lesson Learned – Developing Residue Analytical Methods for Triazole (Conazole) Pesticides in Crop Matrices

Recently, analytical methods were developed in our lab to determine the residues of a parent and two of its major metabolites for a triazole compound in crop and animal matrices. Acidic hydrolysis is commonly included in the triazole residue methods to cleave the conjugated metabolites in biological sample matrices. It is also found that some of the triazole parent and metabolites were all converted to common moieties during hydrolysis. In this case, the total (combined) residues as parent equivalent were normally determined and reported. However, most recently for the global harmonization of MRLs to facilitate the international trade, the parent residues alone are also required to be reported, so that the parent residues are used for MRLs and the total residues for dietary risk exposure analysis. Therefore, the residue methods have to be modified to determine the parent residues prior to hydrolysis. An example of method modifications to allow determination of parent only and the total residues will be presented. Additionally, residues of the three triazole common metabolites [triazole (T), triazole alanine (TA), and triazole acetic acid (TAA)] are required to be determined and reported. Risk assessment of those three triazole common metabolites is currently being evaluated by EPA. Data submission of any existing or newly developed triazole compounds need to include the residues of those three triazole common metabolites as well. The number of crop or animal samples for T, TA and TAA residues could be reduced following a specific guideline. Detailed discussions of triazole common metabolites including the residue methods will be provided.