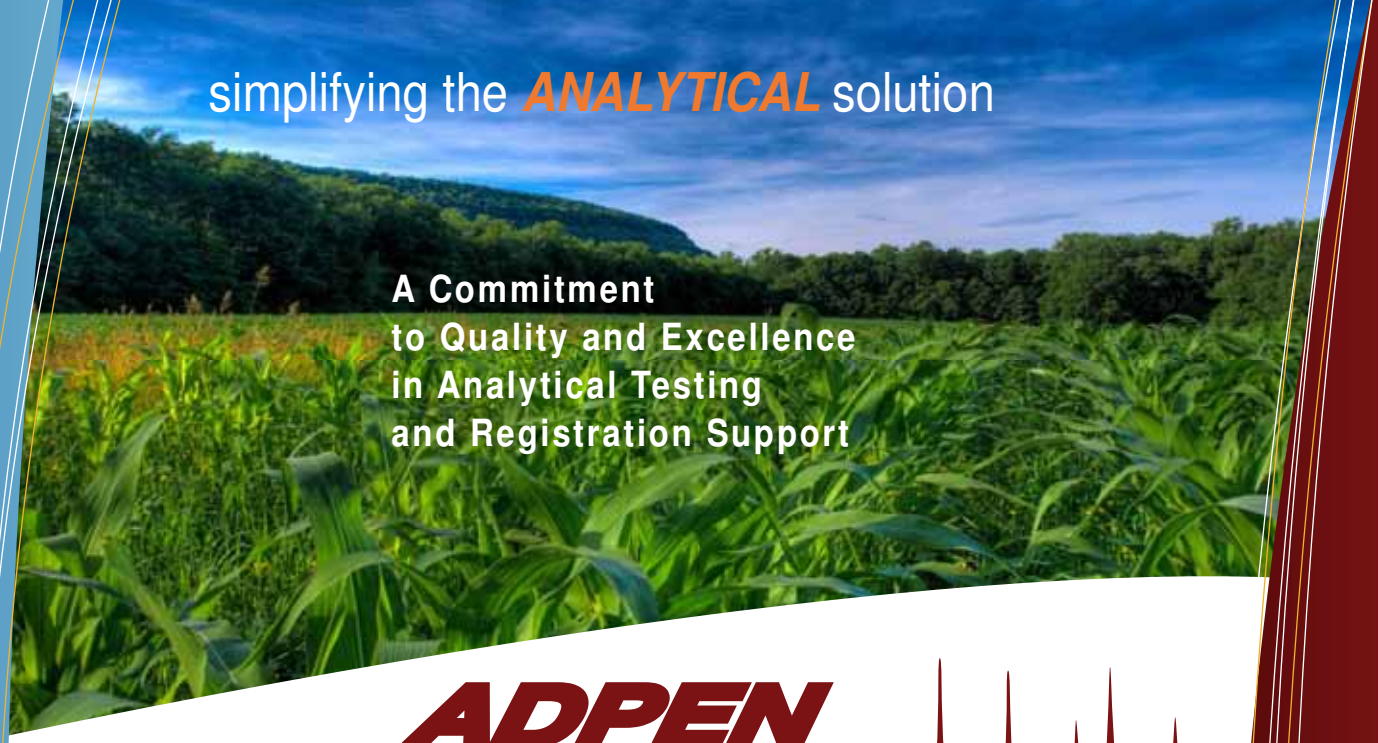
49th Annual
Florida Pesticide Residue Workshop
North American Chemical Residue Workshop



TradeWinds Island Grand Resort
St. Pete Beach, Florida
July 15-18, 2012

"Bringing Scientists Together to Develop and Validate Better Methodologies"

photo courtesy of Visit St. Pete/Clearwater



ADPEN LABORATORIES INC.





ADPEN Laboratories in Jacksonville, FL provides contract analytical services to fill all your testing needs. Our residue testing experience since 1989 means we are the right independent GLP Lab with a commitment to Quality and Excellence. ADPEN has a professional staff and utilizes the latest cutting-edge instrumentation, including the latest LC-MS/MS, GC and GC/MS. Our fleet of LC-MS/MS instrumentation include Agilent's 6490 and ABSciex's 5500.

Our clients have experienced firsthand ADPEN's commitment to provide the absolute best in analytical testing and registration support. We understand our clients are working hard to protect our environment and food supply, and making our world a better place. ADPEN has a track record of providing the simplified analytical solutions our clients need to succeed.

*After more than 20 years, **ADPEN** is still the Laboratory you can Trust!*

ADPEN LABORATORIES INC.
11757 Central Parkway
Jacksonville, FL 32224
904.645.9169 **phone**
904.641.8423 **fax**
www.ADPEN.com

Come see us at:

BOOTH#7A

Or follow us @AdpenLabs for a chance to win a special beach prize!



Table of Contents

FLAG Works, Inc., 2012 Organzing Committee and 2012 Program Committee.....7

Hotel Floor Plans.....8-9

Sponsors.....10

Meeting at a Glance.....12

General Information.....14-15

Exhibitor and Poster Floor Plan.....16

Exhibitor Listing.....17

Short Course, July 14-15, 2012.....18

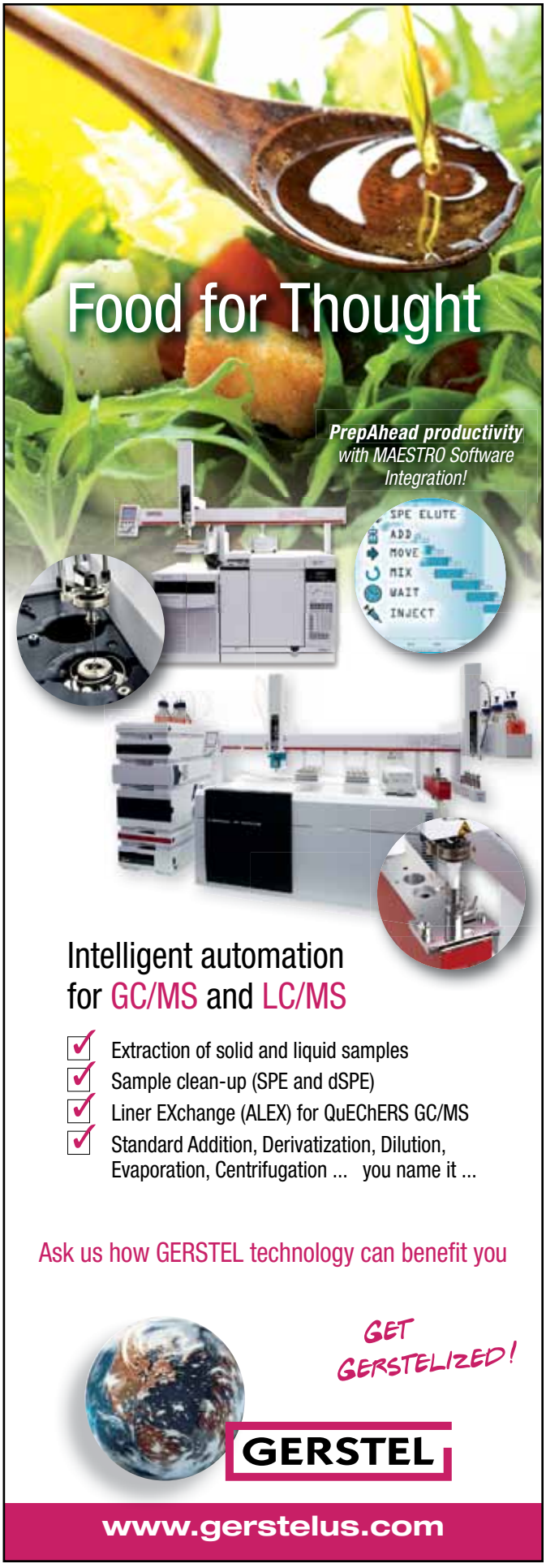
Vendor Seminars.....20
(pre-registration is required)

Meeting Program.....24

Posters (overview).....30

Oral Abstracts.....36

Poster Abstracts.....46



2012 FLORIDA PESTICIDE RESIDUE WORKSHOP
North American Chemical Residue Workshop

July 15, 2012

Dear Attendees and Exhibitors,

Welcome to the 49th Annual Florida Pesticide Residue Workshop. We extend our greetings to long-time attendees and first time attendees as well as our international guests. We also thank our Exhibitors for their generous support of the Workshop. Effective for 2013, FPRW will become the North American Chemical Residue Workshop. Although the name may change, the Organizing and Program Committees will continue to provide interesting scientific sessions and events that stimulate discussion and our further our knowledge in residue analysis. Do not forget to cast your vote for the new logo!

As a follow up to last year's LC/MS/MS techniques short course, we are offering a two-day (Saturday and Sunday) short course on interpretation of MS/MS spectra generated by LC/MS taught by Professor O. David Sparkman. I hope you are able to take advantage of this unique learning experience.

This year we are also offering a dinner cruise on Monday evening. Join your friends and colleagues and enjoy an evening of dinner, drinks, and dancing with a live band aboard the Starlite Majesty. This is a great opportunity to network and experience the scenic views of the inlet waters of Boca Ciega Bay.

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

We have a fantastic three day program lined up for you! Monday morning session starts with Current Topics in Residue Analysis followed in the afternoon by Novel Analysis of Pesticides and Veterinary Drug Residues. Tuesday we start with Global Trade Issues/International Monitoring and in the afternoon we have Southern US State Laboratories Report Their Chemical Residue Activities: Doing More with Less. Wednesday begins with Mega Methods and HRMS Data followed by Residues in the Environment and ending with "Cream of the Crop Technical Talks."

For the poster session this year we requested a vertical format and the majority of the over 100 poster submissions have complied. This enables all of the posters to be displayed for the entire meeting. Please visit the posters, speak to the authors and vote for the best poster. The Journal of Agricultural and Food Chemistry is again offering to publish a special edition affiliated with FPRW. If you have a manuscript to submit for publication, please inform Sherri Turnipseed, FPRW 2012 Program Chair.

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

The FPRW Organizing Committee Planning Meeting takes place on Tuesday evening from 5:00 to 6:00 PM. FPRW is organized by volunteers who participate in the Organizing and Program Committees. Please join us for this year's meeting, get involved and help us continue an outstanding meeting. FPRW is sponsored by FLAG Works, Inc., a not-for-profit organization, which has an agreement with ACS (via the AGRO Division) to help plan and coordinate the event.

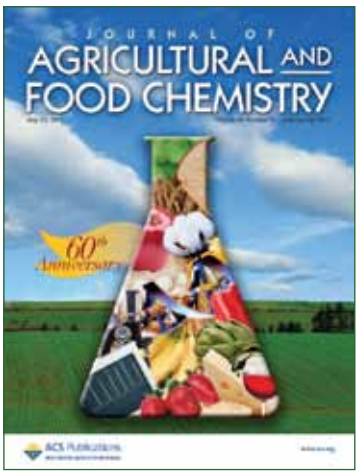
Finally, we would like to thank the 2012 Organizing Committee and the Program Committee, and all of the volunteers for arranging and participating in a great meeting. Enjoy!

Joe Romano, Sherri Turnipseed, Nina Huffstetler, Loretta Fourrier, Mike Telepchak, Phil Wylie, Kate Mastovska, Alaa Kamel, Lynda Podhorniak , Steven Lehotay (2012 Organizing and Program Committee Officers)

JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY

60th Anniversary

The Leader in Agriculture, Food Science & Technology, and Applied Chemistry



INTERESTED IN MORE PESTICIDE CHEMISTRY RESEARCH?

View research on pesticide chemistry and other related topics in the **Symposium: Florida Pesticide Residue Workshop 2011** issue published by the *Journal of Agricultural and Food Chemistry* at **pubs.acs.org/jafc**.

FEATURED ARTICLES FROM THE SYMPOSIUM: FLORIDA PESTICIDE RESIDUE WORKSHOP 2011 ISSUE

Pesticide Residues in Imported, Organic, and "Suspect" Fruits and Vegetables
Carl K. Winter
DOI: 10.1021/jf205131q

Analysis of Veterinary Drug Residues in Frog Legs and Other Aquacultured Species Using Liquid Chromatography Quadrupole Time-of-Flight Mass Spectrometry
Sherri B. Turnipseed, Susan B. Clark, Joseph M. Storey, and Justin R. Carr
DOI: 10.1021/jf2049905

Occurrence and Distribution Study of Residues from Pesticides Applied under Controlled Conditions in the Field during Rice Processing
Lucía Pareja, Marcos Colazzo, Andrés Pérez-Parada, Natalia Besil, Horacio Heinzen, Bernardo Böcking, Verónica Cesio, and Amadeo R. Fernández-Alba
DOI: 10.1021/jf205293j

Insecticide Residues in Pollen and Nectar of a Cucurbit Crop and Their Potential Exposure to Pollinators
Galen P. Dively, Alaa Kamel
DOI: 10.1021/jf205393x

Effect of Sampling Size on the Determination of Accurate Pesticide Residue Levels in Japanese Agricultural Commodities
Masahiro Fujita, Tomonari Yajima, Kazuaki Iijima, and Kiyoshi Sato
DOI: 10.1021/jf3001923

ACS Publications
MOST TRUSTED. MOST CITED. MOST READ.

Screen More Often



Use DART® MS
Detect Pesticides in Seconds

Learn more at
ionsense.com/pesticides

ionSense®
Your source for OpenSpot mass spectrometry

999 Broadway | Suite 404 | Saugus, MA 01906 | (781) 484-1043 | info@ionsense.com

FLAG Works, Inc. / Florida Pesticide Residue Workshop

Board of Directors

J. D. Warren, Chair
Pat Beckett
Susan Eigen
Sherry Garris
Danny LeCompte
Frank Schenck

Executive Director
Teri Besse

Organizing Committee

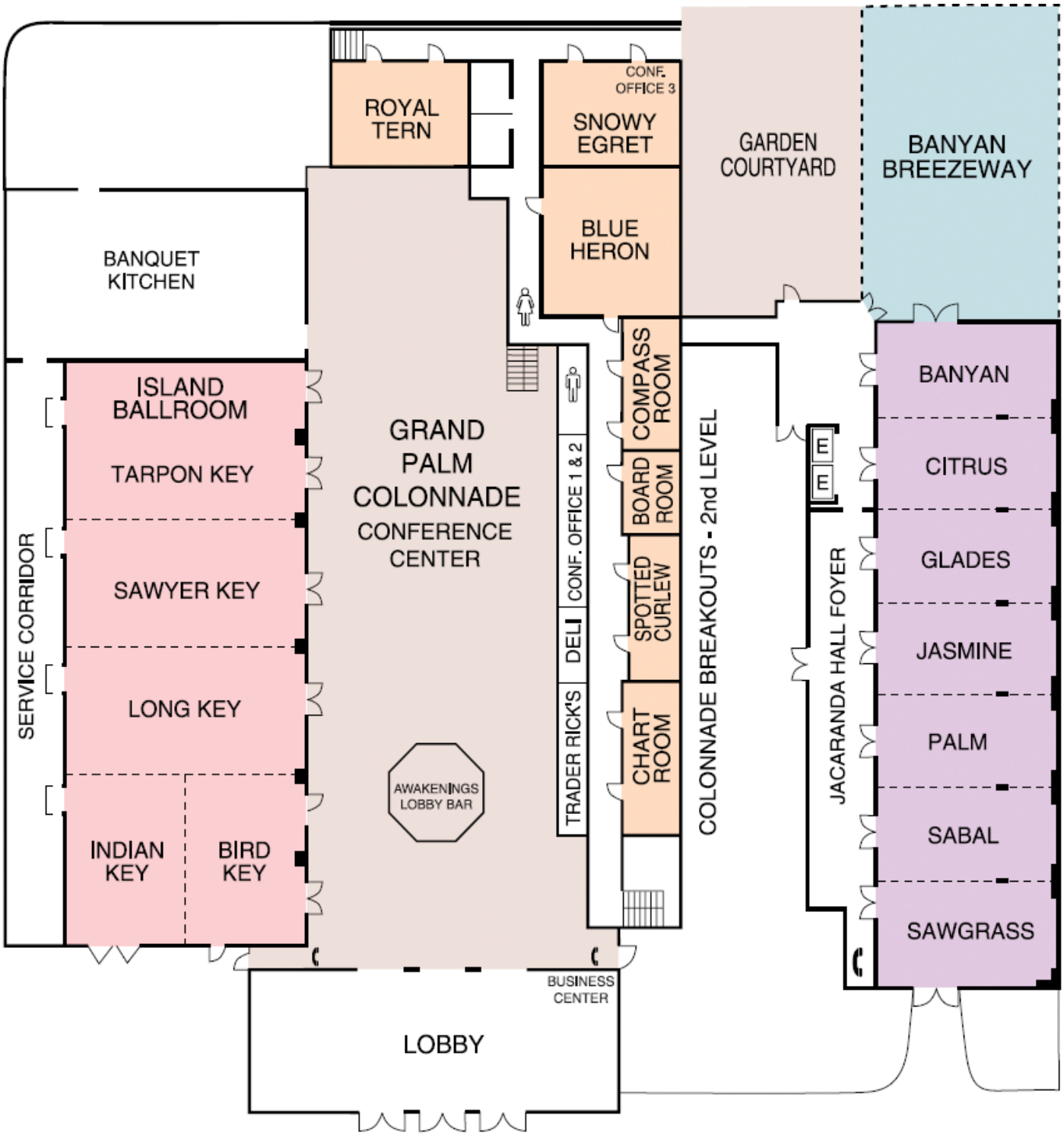
President:	Joe Romano, Waters Corporation
Secretaries:	Loretta Fourrier, LSU Ag Chemistry Laboratory/LSU Ag Center Nina Huffstetler, Louisiana Department of Agriculture and Forestry
President-Elect:	Michael Telepchak, UCT, Inc.
Immediate Past President:	Phil Wylie, Agilent Technologies, Inc.

Program Committee

Chair:	Sherri Turnipseed, US Food and Drug Administration
Chair-Elect:	Kate Mastovska, Covance Laboratories
Immediate Past Co-Chair:	Alaa Kamel, US Environmental Protection Agency
Immediate Past Co-Chair:	Lynda Podhorniak, US Environmental Protection Agency
2010 Chair:	Steve Lehotay, USDA Agricultural Research Service

Program Committee Members
Kevin Armbrust – Mississippi State University
Brad Barrett – AB Sciex
Amy Brown – Florida Department of Agriculture and Consumer Services
Jo Marie Cook – Florida Department of Agriculture and Consumer Services
Jack Cochran – Restek Corporation
Johannes Corley – Rutgers University
André de Kok, NVWA - Netherlands Food and Consumer Product Safety Authority, The Netherlands
Sherry Garris – South Carolina Department of Agriculture
Julie Kowalski –Restek Corporation
Perry Martos – University of Guelph
Chris Sack – US FDA/Total Diet Research Center
Charles Stafford – EPA Analytical Chemistry Branch
Jon Wong – US FDA/Center for Food Safety and Applied Nutrition
Paul Yang –Ontario Ministry of the Environment
Donglei Yu – US FDA/Center for Veterinary Medicine
Larry Zintek – EPA Chicago Regional Laboratory


GULF OF MEXICO
BEACH
W
S
N
E
Cabana Hut
South Beach Lawn
Jacaranda Beach
Flying Bridge
SeaBreeze Terrace
Conclave
Poinciana
Salty's Beach Bar
ADULT POOL
Breck Deck North
Bermudas
Beef 'O' Brady's
Pizza Hut Ice Cream
Body Works Salon
Breckenridge Bldg.
Horizons West
East
Beakers Tropical Outfitters
Fitness Center
Portico
VENDOR SEMINARS
Allamanda Bldg.
Jacaranda Bldg.
POOL & HOTTUB AREA
Hibiscus
Hibiscus
Garden Courtyard
Banyan Breezeway
Pet Play Zone
Sharktooth Tavern
Parking
Palm Court
POOL AREA
TAZ Activities
Tennis Courts
The Pavilion
Parking
Island Ballroom
Grand Palm Colonnade CONFERENCE CENTER
Jacaranda Hall
Paddleboat Landings
Pirate Island
South Parking
Cypress Villa
North Parking
EXHIBITS AND POSTERS
Parking
Magnolia Offices
Admin Offices (5600)
Lobby
REGISTRATION DESK AND MEETING ROOM
Parking
HERTZ CAR RENTAL
GULF BLVD.
TRADEWINDS SANDPIPER SUITES



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

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

Platinum Sponsors

Agilent Technologies

BRUKER

RESTEK

UCT

Waters

THE SCIENCE OF WHAT'S POSSIBLE.™

Host Sponsor

VISIT St. Petersburg Clearwater

VisitStPeteClearwater.com

LAPRW 2013

4° Congreso Latinoamericano sobre Residuos de Plaguicidas

alimentos y medio ambiente

4th Latin American Pesticide Residue Workshop

Food and environment

Fecha límite para recepción de resúmenes
- Oral: 18 de Febrero de 2013
- Poster: 30 de Marzo de 2013

Mayo 26-29 2013
May 26-29 2013
Bogotá - Colombia

Abstracts submission dead line
- Oral: February 18th, 2013
- Poster: March 30th, 2013

www.laprw2013.com
laprw2013_fcbog@unal.edu.co

Organizado por
Organized by

UNIVERSIDAD NACIONAL DE COLOMBIA
SEDE BOGOTÁ





MEETING AT A GLANCE

Saturday, July 14, 2012		
8:00 am - 4:00 pm	**Short Course- O. David Sparkman**	**Sabal/Sawgrass**
Sunday, July 15, 2012		
8:00 am - 3:00 pm	**Short Course- O. David Sparkman**	**Sabal/Sawgrass**
4:00 – 6:00 pm	FDA/State Round Table	Jasmine/Palm
3:00 – 7:00 pm	Registration	Grand Palm Colonnade
3:00 – 6:00 pm	Exhibitor Setup	Pavilion
3:00 – 6:00 pm	Poster Board Setup	Pavilion
6:00 pm	Technical Session Setup	Ballrooms
6:30 – 6:45 pm	Moderator Training	Ballrooms
6:30 – 7:30 pm	**Restek Evening Seminar**	**Horizon Rooms**
8:00 – 9:00 pm	**Thermo Scientific Evening Seminar**	**Horizon Rooms**
Monday, July 16, 2012		
7:15 – 8:15 am	Early Morning Coffee	Grand Palm Colonnade
7:30 – 5:00 pm	Registration	Grand Palm Colonnade
7:30 – 10:30 am	Exhibitor Setup	Pavilion
7:30 – 10:30 am	Poster Board Setup	Pavilion
7:15 – 8:15 am	**Waters Breakfast Seminar**	**Horizon Rooms**
8:30 – 10:45 am	Current Topics in Residue Analysis	Ballrooms
10:45 – noon	Exhibition Opening	Pavilion
11:00 – noon	Poster Session A	Pavilion
12:15 – 1:15 pm	**LECO Lunch Seminar**	**Horizon Rooms**
1:30 – 2:45 pm	Novel Analysis of Pesticides	Ballrooms
2:45 – 3:30 pm	BREAK (Exhibition & Posters)	Pavilion
3:30 – 5:10 pm	Veterinary Drug Residues	Ballrooms
6:00 pm	Depart for Dinner Cruise!	TradeWinds Hotel Entrance
10:00 pm	Return from Dinner Cruise	Dock where Buses parked
Tuesday, July 17, 2012		
7:30 – 4:30 pm	Registration	Grand Palm Colonnade
All Day	Exhibition & Posters	Pavilion
7:15 – 8:15 am	Early Morning Coffee	Pavilion
7:15 – 8:15 am	**AB SCIEX Breakfast Seminar**	**Horizon Rooms**
8:30 – 10:35 am	Global Trade Issues/International Monitoring	Ballrooms
10:35 – noon	BREAK (Exhibition & Posters)	Pavilion
11:00 – noon	Poster Session B	Pavilion
12:15 – 1:15 pm	**UCT Lunch Seminar**	**Horizon Rooms**
1:30 – 3:00 pm	State Laboratories: Doing More with Less	Ballrooms
3:00 – 3:45 pm	BREAK (Exhibition & Posters)	Pavilion
3:45 – 5:00 pm	Forum on Mass Spectrometry	Ballrooms
5:00 – 6:00 pm	Organizing Committee Meeting	Ballrooms
7:00 – 8:00 pm	Reception by TradeWinds	Pavilion
8:00 pm →	Beach Volleyball Game	On the Beach
Wednesday, July 18, 2012		
7:30 – 12 noon	Registration	Grand Palm Colonnade
Until noon	Exhibition & Posters	Pavilion
7:15 – 8:15 am	Early Morning Coffee	Pavilion
7:15 – 8:15 am	**Agilent Breakfast Seminar**	**Horizon Room**
8:30 – 10:35 am	Mega Methods and HRMS Data	Ballrooms
10:35 – noon	BREAK (Exhibition & Posters)	Pavilion
11:00 – noon	Poster Session C	Pavilion
12:15 – 1:15 pm	**BUCHI Lunch Seminar**	**Horizon Rooms**
1:30 – 2:45 pm	Residues in the Environment	Ballrooms
2:45 – 3:00 pm	BREAK	Grand Palm Colonnade
3:00 – 4:40 pm	"Cream of the Crop Technical Talks"	Ballrooms
4:40 – 5:00 pm	Closing Ballrooms	
	Poster Awards and NACRW Logo Selection!	

waters.com



THE STRONG, SENSITIVE TYPE.

Introducing Xevo G2-S QTof.

Combine revolutionary, high sensitivity StepWave™ technology with durability that goes the distance, and you've got an accurate mass MS/MS benchtop system like no other. With Xevo® G2-S QTof, scientists can now detect compounds at levels never before possible on a benchtop Tof instrument. At the same time, you're maintaining the highest UPLC® performance. This is mass spectrometry at it's very best. **To learn more, visit waters.com/xevog2sqtof.**

Waters

THE SCIENCE OF WHAT'S POSSIBLE.™

Pharmaceutical & Life Sciences | Food | Environmental | Clinical | Chemical Materials

©2012 Waters Corporation. Xevo and UPLC are registered trademarks of Waters Corporation. StepWave and The Science of What's Possible are trademarks of Waters Corporation.

GENERAL INFORMATION

Registration

Check in once at the registration desk at your earliest opportunity
Sunday - 3:00 – 7:00 pm
Monday - 7:30 am – 5:00 pm
Tuesday - 7:30 am – 4:30 pm
Wednesday - 7:30 am – Noon

KEY to Presentation Numbering System

Oral presentations are numbered O-1, O-2, O-3, O-4, etc.
Vendor Seminars are numbered V-1, V-2, V-3, V-4, etc.
Session A posters are ODD numbered P-1, P-3, P-5, etc.
Session B posters are EVEN numbered P-2, P-4, P-6, etc.
Session C posters are ALL posters

Poster Sessions (in The Pavilion)

Hang Posters Sunday evening from 3:00 pm to 6:00 pm or before 10:30 am on Monday
Take down posters between 12 noon to 2:00 pm on Wednesday

Posters may be viewed any time Exhibition is open

Poster Session A authors must be at their posters from 11:00 am – noon on Monday
Poster Session B authors must be at their posters from 11:00 am – noon on Tuesday
Poster Session C all authors present at their posters from 11:00 am – noon on Wednesday

Poster Prize

Author(s) of Best Poster will be determined by popular vote of attendees and will win the prize.
Attendees must place their votes in the ballot box by noon on Wednesday. Don't forget to get your ticket to win a prize for voting for the Best Poster.

Exhibition

Monday – 10:45 am - 5:00 pm
Tuesday - 7:30 am - 5:00 pm
Tuesday evening reception with cash bar 7:00 - 8:00 pm ***Sponsored by TradeWinds Island Grand Resort***
Wednesday - 7:30 am – noon

Coffee and Breaks

Coffee will be available 7:15 - 8:15 am each morning in The Pavilion and the Grand Palm Colonnade. There will also be mid-morning and afternoon refreshment breaks each day. The Monday and Tuesday mid-morning and afternoon breaks, as well as the Wednesday mid-morning break, will be served in the Exhibition Hall (Pavillion). On Wednesday afternoon, the break will be served in the Grand Palm Colonnade. ***All Coffee Breaks are co-sponsored by the Visit St. Pete/Clearwater Convention and Visitors Bureau.***

Announcements

Moderators will make general announcements from the podium. If you need to have an announcement made, fill out an announcement form and submit it to Teri Besse. These announcement forms will be available at the registration desk.

Job Placement Bulletin Board

Self-serve message board for those offering or seeking employment or to leave notes for others at the meeting.

Door Prizes

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

Submission of Manuscripts to *Journal of Agricultural and Food Chemistry*

You are encouraged to contribute original research and/or review articles to the *Journal of Agricultural and Food Chemistry* for a special section related to FPRW in 2012. Please inform Sherri Turnipseed in advance if you intend to submit an article**.** The manuscripts must be submitted electronically online on the journal's web site by September 1, 2012. Manuscripts will undergo the normal peer-review process to determine if they are acceptable for publication.

Copies of Presentations

Oral Presentations: Following the meeting, if time and resources permit, oral presentations will be posted on our web site if author permission is granted. There are limitations to what we can post. Absolutely no files will be posted without a speaker's written permission (historically, two thirds of our speakers have given permission). The Power Point files are converted to PDF format, 2 slides per printed page. The file conversion is necessary due to limited server space (the file size of PDF format is roughly 10-20% that of PPT format). Various security restrictions may be added to the PDF file per speaker's request (such as disabling "copy text" and "print" functions). Some slides containing confidential or proprietary information may be deleted.

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

Attendee List

After the meeting (approximately two weeks) an attendee list will be available, but only by request. Please contact Teri Besse at teribesse@comcast.net.

Meeting Website

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

Meeting Evaluations

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

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

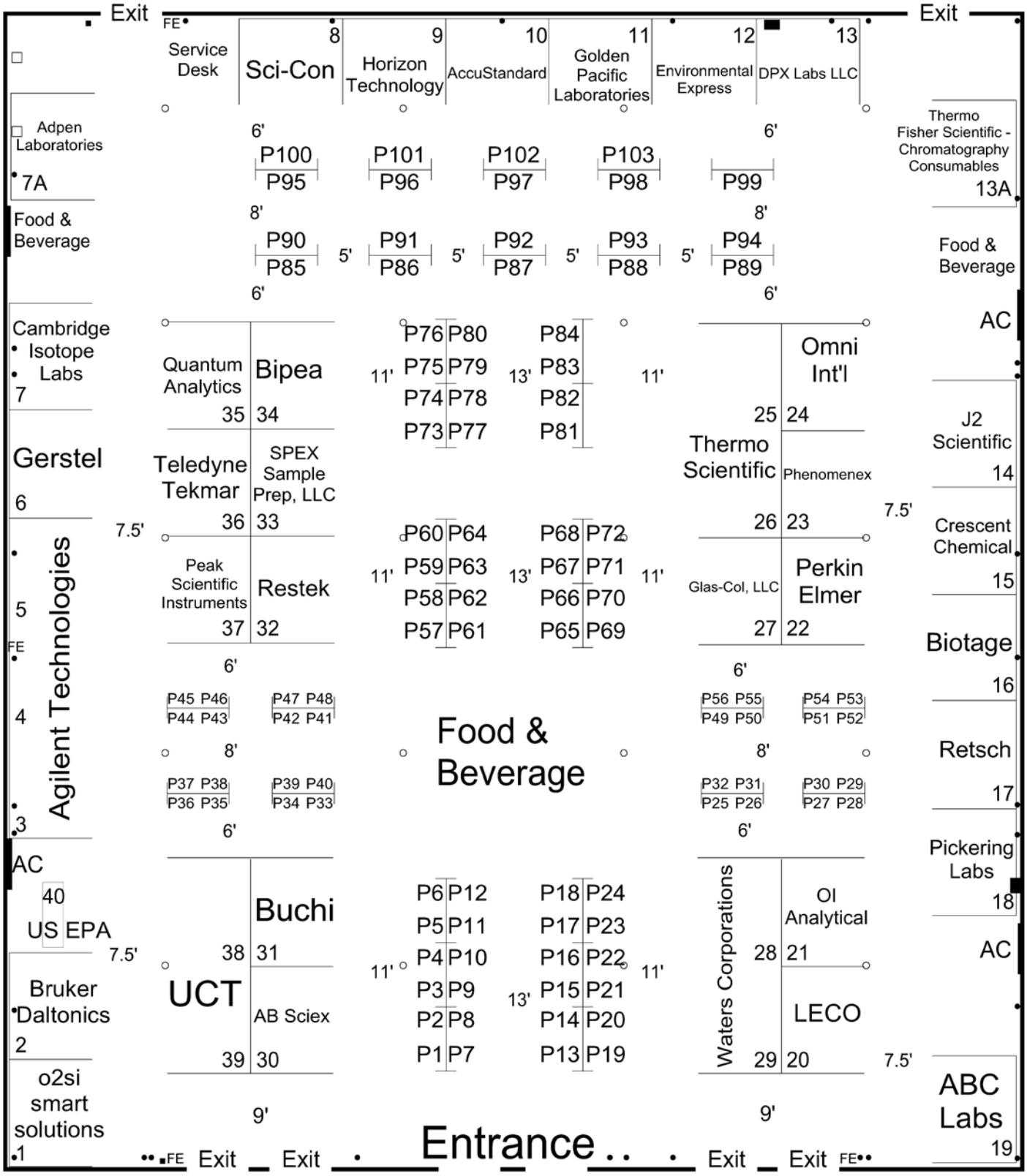
FUTURE MEETING DATES

2013 July 21 - 24
2014 July 20 - 23
2015 July 19 - 22

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

Exhibits and Poster Sessions

Location: Pavillion



EXHIBITORS

AB SCIEX
Booth #30
www.absciex.com

ABC Laboratories, Inc.
Booth #19
www.abclabs.com

Adpen Laboratories, Inc.
Booth #7A
www.adpen.com

Agilent Technologies
Booth #3,4,5
www.agilent.com/chem

AccuStandard
Booth #10
www.accustandard.com

Bipea
Booth #34
www.bipea.org

Biotage
Booth #16
www.biotage.com

Bruker Daltonics
Booth #2
www.bdal.com

BUCHI Corporation
Booth #31
www.mybuchi.com

Cambridge Isotope Labs
Booth #7
www.isotope.com

Crescent Chemical Co Inc.
Booth #15
www.crescentchemical.com

DPX Labs, LLC
Booth #13
www.dpxlabs.com

Environmental Express, Inc.
Booth #12
www.envexp.com

Glas-Col, LLC
Booth #27
www.glascol.com

Gerstel, Inc.
Booth #6
www.gerstelus.com

Golden Pacific Laboratories
Booth #11
www.gplabs.com

Horizon Technology Inc.
Booth #9
www.horizontechinc.com

J2 Scientific
Booth #14
www.j2scientific.com

LECO Corporation
Booth #20
www.leco.com

o2si smart solutions
Booth #1
www.o2si.com

OI Analytical
Booth #21
www.oico.com

OMNI International, Inc.
Booth #24
www.omni-inc.com

PEAK Scientific Instruments LTD
Booth #37
www.peakscientific.com

Perkin Elmer
Booth #22
www.perkinelmer.com

Phenomenex
Booth #23
www.phenomenex.com

Pickering Laboratories, Inc.
Booth #18
www.pickeringlabs.com

Quantum Analytics
Booth #35
www.lqa.com

Restek Corporation
Booth #32
www.restek.com

Retsch Inc.
Booth #17
www.retsch-us.com

SCICON/FMS
Booth #8
www.scicon.net and
www.fmsenvironmental.com

SPEX Sample Prep
Booth #33
www.spexsampleprep.com

Teledyne Tekmar
Booth #36
www.tekmar.com

Thermo Scientific
Booth #25,26
www.thermoscientific.com

Thermo Fisher Scientific - Chromatography Consumables
Booth #13A
www.separatedbyexperience.com

UCT
Booth #38,39
www.unitedchem.com

U.S Environmental Protection Agency
Booth #40
www.epa.gov

Waters Corporations
Booth #28,29
www.waters.com

SHORT COURSE

Saturday, July 14, 2012 8:00 am to 4:00 pm
Sunday, July 15, 2012 8:00 am to 3:00 pm

Location: Sabel and Sawgrass Rooms

Interpretation of MS/MS spectra generated by LC/MS

Instructor: Professor O. David Sparkman

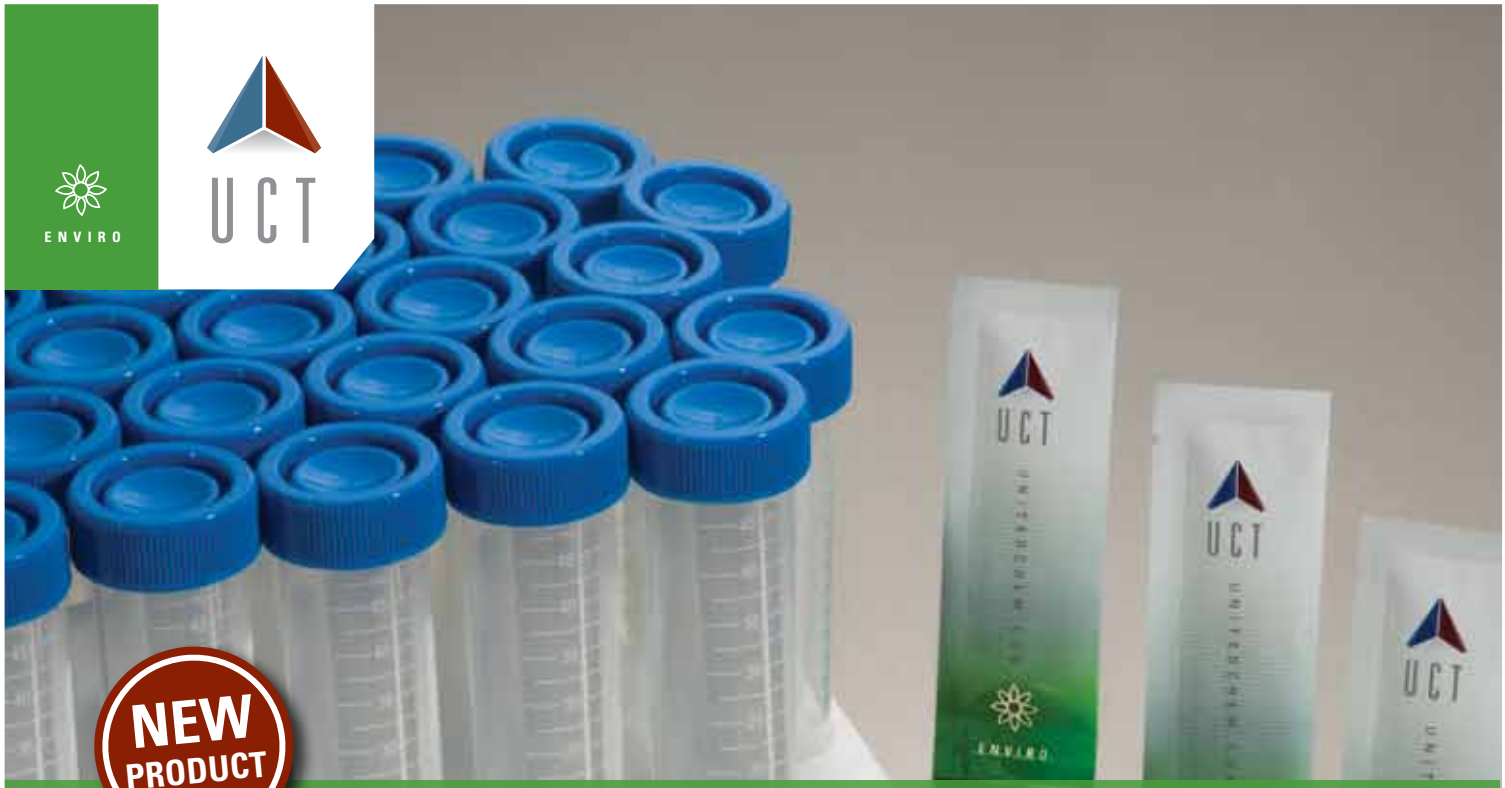
This course will cover the determination of elemental composition from mass spectral data using accurate mass, isotope-peak intensity ratios, and the Nitrogen Rule. Structure of ions will be supported through fragmentation mechanisms. Strategies involving the use of the three different types of MS/MS (tandem-in-space) to confirm structure will be explained. Example data will be interpreted and attempts will be made to interpret participant-supplied data.

About the Instructor

O. David Sparkman is a consultant in mass spectrometry and an instructor of mass spectrometry short courses for the American Chemical Society (ACS). He has been active in the field of analytical chemistry since 1964 and has been actively involved with mass spectrometry and hyphenated techniques in mass spectrometry since that time. Prior to becoming a consultant, he spent several years in the commercial mass spectrometry manufacturing field holding positions in product development, applications research, and marketing. His current consulting includes work with the National Institute of Standards and Technology on search algorithms for mass spectral databases and the use of databases of electron ionization spectra in the interpretation of spectra obtained through collisionally activated dissociation. He currently is the director of six different ACS courses in mass spectrometry, holds positions on the Editorial Advisory Boards of the Journal of the American Society for Mass Spectrometry and the HD Science GC/MS Update – Part B, is on the continuing education committee of the ACS, and is a frequent contributor of conference and book reviews to the Journal of the American Society for Mass Spectrometry. He also has a number of clients among research organizations conducting applications and instrument development research in mass spectrometry, as well as consulting for instrument manufacturers.

ENVIRO

UCT



NEW PRODUCT

UCT ENVIRO-CLEAN® QuEChERS MULTI-PACKS

- Introducing new convenience to QuEChERS analysis
- The UCT ENVIRO-CLEAN® QuEChERS Mylar Mini-Pouch makes the method even easier to use by simplifying the addition of reagents
- The pouch, with its convenient tear notch and narrow design, easily fits into a 50 mL centrifuge tube making transfer of reagents efficient and assured



MADE IN USA

800.541.0559 www.unitedchem.com

VENDOR SEMINARS

Food and beverage provided by each company
Pre-registration is required for all Vendor Seminars.
Please sign up at the meeting registration desk

V-1 Sunday Evening, July 15, 2012 6:30 pm to 7:30 pm Restek

Location: Horizon Room

How Dirty are You? Common Lab Practices that may Impact Data Quality are Examined Using Various Gas Chromatography and Gas Chromatography-Mass Spectrometry Techniques

Julie Kowalski, Restek Corporation, 110 Benner Circle, Bellefonte, PA

For anyone who ever wondered if your lab mates are conscientious or crazy, we put common lab practices and their perversions to the test. Does your colleague madly rinse syringes to avoid carryover? 2 times? 10 times? How many times do you rinse? Do you work with someone who is obsessed with finger oil and becomes unhinged when you touch a vial cap or GC inlet liner? Do you tell incredible tales of contamination after using the wrong solvent with pipette tips? Have you had a co-worker call you from home asking you to recap their punctured sample vials?

Using GC and GC/MS technology, we methodically examine the effects of common lab procedures on the cleanliness of sample and instrument systems. We test syringe rinsing, sample exposure to septa, pipette tip/solvent combinations and other everyday procedures. We tested the handling skills of our chemists who range from five to twenty five years of experience and found out which are the dirtiest. Attend this Restek seminar to see how dirty you are based on our results and experimentally determined best lab practices. Join us for dinner and the “How dirty am I?” quiz to win prizes.

V-2 Sunday Evening, July 15, 2012 8:00 pm to 9:00 pm Thermo Scientific

Location: Horizon Room

Multiresidue Pesticides Analysis Using UHPLC-Orbitrap Mass Spectrometry: Method Development, UHPLC Parameters, Mass Accuracy and Method Performance

Dr. Paul Yang, Ontario Ministry of the Environment, Ontario, Canada

Multiresidue methods are employed to monitor chemical contaminants in environmental and food samples and to ensure that concentrations present do not exceed the tolerance levels established by government regulations. Ultrahigh performance liquid chromatography-high resolution mass spectrometry (UHPLC-HRMS) based multiresidue methods have been the focus of much research since the turn of the century. The goal of this work was to develop and evaluate the UHPLC-orbitrap mass spectrometry system (UHPLC-Orbitrap) based multiresidue method for automated, targeted analysis of pesticides in environmental and food matrices. Analytical data were acquired using pesticide standards of seven concentrations, four mass resolving power settings and in replicate analysis (eight) in solvent and sample matrices. These data were used to determine instrument detection limits (IDL), matrix specific method detection limits (MDL) and the ability for automated identification of targeted pesticides. The same data also demonstrated that optimized Orbitrap-MS extracted ion chromatogram (XIC) data can be collected at a resolving power of 70,000 or higher as long as there is a minimum of 5–7 data points to define a specific chromatographic peak. Examples of the use of various UHPLC parameters to determine the root cause of mass accuracy during the analysis are presented.

How Applying Smarter GC-Triple Quadrupole Software to Pesticide Analysis Can Improve Your Results

Eric Phillips, Senior Global Applied Markets Manager, Thermo Fisher Scientific

Over recent years, GC-triple quadruple MS has increased in popularity due to its ability to offer lower detection limits in complex matrices, simplified sample prep requirements, and faster analysis times. But with new instrument technology comes the need for new skills to harness the advantages of this new technology. Two new challenges to using GC-triple quadrupole for a complex analysis such as pesticides in food, is determining the selected reaction monitoring (SRM) transitions that make up a typical MS/MS method, and maintaining the acquisition windows of such a method when a large number of compounds are being monitored. In this presentation, a strategy for addressing both of these challenges is discussed in the context of new software designed to automate common method development and method maintenance tasks. Also, in addition to making the triple quadrupole easier to use, this strategy can increase sensitivity of the analysis, which will be demonstrated using a complex SRM pesticide method as an example.

V-3 Monday Morning, July 16, 2012 7:15 to 8:15 am Waters Corporation
Location: Horizon Room

One Peak, Many Possibilities
Séverine Goscinny, Wetenschappelijk Instituut Volksgezondheid, Scientific Institute of Public Health

The number of applications of full spectra mode acquisition for monitoring of pesticides in food has rapidly increased. This is the direct consequence of having to efficiently target increasing number of compounds in one run. Although, time-of-flight analyzers are well suited for this task, their analytical power can be enhanced in presence of large amounts of interference which result mainly from the matrix and less so from solvents and reagents. What if you had an analytical tool allowing you to filters your spectrum from these interferences and at the same time offering the potential of unambiguous identification of your pesticides?

Ion mobility-mass spectrometry with MSE mode offers three distinct information identification points (drift time, precursor and fragment ions) under one chromatographic peak plus a unique feature of the mobility to clean the spectrum by resolving the target ions from interferences. Therefore, analysis performed on a Waters Synapt in mobility mode enables the analyst to have more confidence in his results. During this talk, we will demonstrate how to integrate these different tools in a screening method for pesticide residues in food.

V-4 Monday Lunchtime, July 16, 2012 12:15 to 1:15 pm LECO
Location: Horizon Room

From Dioxins to Pesticides to Pharmaceutical Compounds - Comprehensive and Targeted Application of High Resolution Time of Flight Mass Spectrometry in Food and Environmental Analysis
Doug Hayward, USFDA and Jeffrey S. Patrick, LECO Separation Science

Environmental analyses require instrumentation and techniques which meet stringent demands. High resolution time of flight mass spectrometry (HRTOF) meets many of these demands. The utility of HRTOF to address selectivity issues not approached through the use of triple quadrupoles will be discussed. Specifically, the analysis of pesticides and related compounds benefits from the high resolution and mass accuracy offered by HRTOF. Both targeted and comprehensive analysis of the pesticide content will be shown in a variety of matrices including ginger and black cohosh for both GC and HPLC interfaced to HRTOF. Novel tools for data processing including targeted methods leveraging isotopes, fragment ions and the correlation of precursor/fragment ion pairings will demonstrate advantages to interrogating environmental data. One of the most challenging issues is dioxin analysis to meet EPA 1613b standards while having the ability to obtain comprehensive data on a single platform. The ability to detect dioxins at levels surpassing EPA 1613b criteria using GC interfaced to high resolution time of flight mass spectrometry will be demonstrated. Data on other POPs and halogenated compounds acquired in the same samples on the same instrument will be discussed to demonstrate the versatility in meeting broad needs.

V-5 Tuesday Morning, July 17, 2012 7:15 – 8:15 am AB SCIEX
Location: Horizon Room

Advanced Tools to Screen for Known and Unknown Chemical Residues in Food Samples
André Schreiber, AB SCIEX, Concord, ON (Canada)

Liquid Chromatography coupled to tandem Mass Spectrometry (LC-MS/MS) is a powerful analytical tool for the analysis of polar, semi-volatile, and thermally labile compounds of a wide molecular weight range, such as pesticides, veterinary drugs, mycotoxins and other food residues. Mass analyzers based on triple quadrupole technology operated in Multiple Reaction Monitoring (MRM) mode deliver highly selective and sensitive quantitative results and are therefore well established for multi-target screening of food contaminants. Compound identification is typically performed by monitoring of two MRM transitions and calculating the area ratio of quantifier and qualifier ion. The potential risk of false positive and negative results can be further minimized by MS/MS library searching using a QTRAP® hybrid LC/MS/MS system. MS/MS spectra contain the complete molecular fingerprint of a molecule and thus providing an added degree of confidence for compound identification.

Here we present and compare the application of different LC-MS/MS techniques for targeted and non-targeted pesticide residue screening. A variety of food samples was analyzed for pesticides using triple quadrupole, QTRAP®, and TripleTOF™ systems after QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) extraction.

V-6 Tuesday Lunchtime, July 17, 2012 12:15 to 1:15 pm UCT
Location: Horizon Room

Strong or Weak Ion Exchange
Their Impact on Extraction Outcomes

Mike Telepchak and Brian Kinsella, UCT, 2731 Bartram Rd., Bristol, PA 19007

Various dyes are used in the aquaculture industry necessitating methods for their analysis in fish tissue. Recent inquiries have shown difficulties in eluting certain of the dyes from strong cation exchangers. This presentation looks at the problems encountered, the reason behind the problems and the solutions required for the analysis of the compounds of interest.

V-7 Wednesday Morning, July 18, 2012 7:15 to 8:15 am Agilent Technologies, Inc.
Location: Horizon Room

Improving the Quality of your MS Data by Implementing Sample Preparation Techniques

Joanie Stevens, Agilent Technologies, Inc.

Everyone is very aware of the selectivity and sensitivity offered by LC/MS and GC/MS and tandem MS analysis. However, the analysis of food samples can be extremely challenging because the term “food” encompasses an astronomical range; from a fruit/vegetable to extremely complex components, like breakfast cereals and infant formulations. All foods have some degree of matrix interference, which will directly affect the analysis by MS. The results from compromised samples result in increases in RSDs, higher limits of detection, irreproducible chromatography. Matrix interferences greatly increase maintenance issues as they build up in the system; for instance column and inlet degradation, and source cleaning. Sample preparation will address and eliminate all these issues by removing matrix interferences. Sample preparation is like a toolbox there are many options available, but making a decision can seem overwhelming. However, all one needs to do is evaluate the sample and its matrix; this will literally offer the type of sample preparation that is required. We will discuss this simple approach by examples, and how to offer doable sample preparation techniques to our customers.

Analysis of Known and Unknown Food Contaminants using a New GC/Q-TOF

Phil Wylie, Agilent Technologies, Inc.

Virtually every day brings a new headline reporting a food recall, a contaminated food item, or news about ingredients that may not be safe to eat. With more and more international trade in food and food ingredients, there is an increasing need to monitor food safety. As we learned with the melamine crisis, one never knows what new contaminant might show up in our food. Agilent has designed the world’s only accurate mass high resolution GC quadrupole time-of-flight instrument based on the 7000 GC triple quadrupole MS and the 6500 LC Q-TOF. With full spectral acquisition and accurate mass, this instrument has the ability to “see through” the matrix by extracting characteristic accurate mass ions for the target compounds. One can also calculate the formulas for molecular ions and fragments. MS/MS is used to help identify unknown analytes. Special “Unknowns Analysis” software is used to identify compounds that were not in the target method.

V-8 Wednesday Lunchtime, July 18, 2012 12:15 to 1:15 pm BUCHI Corporation

Location: Horizon Room

Buchi SpeedExtractor – New Dimensions in Pressurized Solvent Extraction for Pesticides and Environmental Contaminants

Susanne Feifel, Product Manager Extraction Solutions, and William Ickes, Product Specialist Extraction Solutions, BUCHI Corporation, 19 Lukens Drive, New Castle, DE 19720

Pressurized solvent extraction is one of the most efficient, modern extraction techniques. With the Buchi SpeedExtractor, you can extract up to 6 samples in parallel in 20 minutes using small volumes of solvent. The sample extracts can then be concentrated in parallel, to a residual volume with the Buchi Syncore Analyst in a fast, environmentally friendly way. Join our seminar and participate in a hands-on demonstration with our Pressurized Solvent Extraction and Parallel Evaporation Instruments. Let us show you first-hand how these instruments can save you time and money for your ever demanding workload.

Designed to work perfectly together, you can perform your extractions quickly with high throughput and eliminate sample transfer by extracting directly into the collection vessels of the Buchi Syncore Analyst. Once the collection vessels are placed on the Buchi Syncore Analyst you can evaporate your samples in parallel, to a residual volume. Finish your sample preparation for pesticides and other environmental contaminants faster than ever with better recoveries due to the unique design of both our extraction and evaporation instruments. Don't miss out on this unique opportunity to participate in a hands-on instrument demonstration!

Easily automate your sample prep with RapidTrace®+

The new Biotage RapidTrace+ delivers advanced processing and rapid development of rugged sample preparation methods. New features accommodate up to 40 mL sample volumes, ideal for water, soil and feed. It is extremely reliable and precise, incorporating easy-to-use features for quick method optimization. The modular design allows you to adjust the capacity to meet your method needs, even if they change over time.

RapidTrace+ has already been successfully implemented in thousands of laboratories worldwide.



Want to learn more?
Come visit us at FPRW 2012, booth #16

Biotage®

MEETING PROGRAM

Saturday, July 14, 2012

8:00 am-4:00 pm	**Short Course – O. David Sparkman**	**Sabal/Sawgrass**

Sunday, July 15, 2012

8:00 am-3:00 pm	**Short Course – O. David Sparkman**	**Sabal/Sawgrass**
4:00 – 6:00 pm	FDA/State Round Table	Jasmine/Palm
3:00 – 7:00 pm	Registration	Grand Palm Colonnade
3:00 – 6:00 pm	Exhibitor Setup	Pavilion
3:00 – 6:00 pm	Poster Board Setup	Pavilion
6:30 – 6:45 pm	Moderator Training	Ballrooms
6:00 pm →	Technical Session Setup	Ballrooms
6:30 – 7:30 pm	**Restek Evening Seminar**	**Horizon Rooms**
V-1	**How Dirty are You? Common Lab Practices that may Impact Data Quality are Examined Using Various Gas Chromatography and Gas Chromatography-Mass Spectrometry Techniques** Julie Kowalski, Restek Corporation, 110 Benner Circle, Bellefonte, PA	
8:00 – 9:00 pm	**Thermo Evening Seminar**	**Horizon Rooms**
V-2	**Multiresidue Pesticides Analysis Using UHPLC-Orbitrap Mass Spectrometry: Method Development, UHPLC Parameters, Mass Accuracy and Method Performance** Dr. Paul Yang, Ontario Ministry of the Environment, Ontario, Canada	
and	**How Applying Smarter GC-Triple Quadrupole Software to Pesticide Analysis Can Improve Your Results** Eric Phillips, Senior Global Applied Markets Manager, Thermo Fisher Scientific	

Monday, July 16, 2012

7:30 – 5:00 pm	Registration	Grand Palm Colonnade
7:15 – 8:15 am	Early Morning Coffee	Grand Palm Colonnade
7:30 – 10:30 am	Exhibitor Setup	Pavilion
7:30 – 10:30 am	Poster Board Setup	Pavilion
7:15 – 8:15 am	**Waters Breakfast Seminar**	**Horizon Rooms**
V-3	**One Peak, Many Possibilities** Séverine Goscinny, Wetenschappelijk Instituut Volksgezondheid, Scientific Institute of Public Health	
8:30 – 10:45 am	**Current Topics in Residue Analysis** *Moderator: Sherri Turnipseed, US FDA*	Ballrooms
8:30 – 8:45 am	**FLAG Works/President, Organizing Committee**	
8:45 – 9:15 am	**Gwendolyn Wyard** – Organic Trade Association, USA	
O-1	**Pesticide Residue Testing Requirements under the National Organic Program**	

9:15 – 9:45 am Bruce Peebles, Catherine Rawlinson, Joel Gummer, Garth Maker, Ian Mullaney, and **Robert Trengove** – Murdoch University, AUSTRALIA
O-2 **Untargeted Residue Analysis and the "omics": The Study Of Health Impacts Of Residue Exposure - A Metabolomics Focus**

9:45 – 10:15 am **Douglas Heitkemper,** Nohora Shockey, Kevin Kubachka, Traci Hanley – US FDA Forensic Chemistry Center; Sean Conklin – US FDA Center for Food Safety and Applied Nutrition, USA
O-3 **Arsenic Speciation in Fruit Juice and Rice Products Using HPLC-ICP-MS**

10:15 – 10:45 am **Ping Wan** – Office of Indiana State Chemist; **Steven Moser** – Oklahoma Dept. of Agriculture, Food, & Forestry, USA
O-4 **2011 Imprelis (Aminocyclopyrachlor) Crisis: What Happened, How We Dealt with the Situation, and Lessons Learned**

10:45 – noon **Exhibition Opening** Pavilion

11:00 – noon **Poster Session A: Authors for ODD number posters present**
Paul Yang and Lawrence Zintek co-coordinators of poster sessions

12:15 – 1:15 pm **LECO Lunch Seminar** **Horizon Rooms**
V-4 **From Dioxins to Pesticides to Pharmaceutical Compounds - Comprehensive and Targeted Application of High Resolution Time of Flight Mass Spectrometry in Food and Environmental Analysis**
Doug Hayward, USFDA and Jeffrey S. Patrick, LECO Separation Science

1:30 – 2:45 pm **Novel Analysis of Pesticides** Ballrooms
Moderators: Lynda Podhorniak & Alaa Kamel, US EPA

1:30 – 1:55 pm **Séverine Goscinny,** Laure Joly, Edwin De Pauw, Vincent Hanot, Gauthier Eppe – Scientific Institute of Public Health, Belgium and University of Liège, BELGIUM
O-5 **Ion Mobility-Mass Spectrometry for the Screening of Pesticides in Food**

1:55 – 2:20 pm **Lawrence Zintek** – US EPA Region 5, USA
O-6 **US EPA UPLC/MS/MS Method Development for Analytes of Concern**

2:20 – 2:45 pm **Rosalie Multari,** David Cremers – Applied Research Associates, Inc., USA; Thomas Scott, Peter Kendrick – Diversified Laboratories, USA
O-7 **The Detection of Pesticides and Dioxins in Oils using Laser-Induced Breakdown Spectroscopy (LIBS)**

2:45 – 3:30 pm **BREAK** (Exhibition & Posters) Pavilion

3:30 – 5:10 pm **Veterinary Drug Residues** Ballrooms
Moderator: Wendy Andersen, US FDA

3:30 – 3:55 pm **Pak-Sin Chu**, Wei Song, Tricia Johnson, Hiranthi Jayasuriya, Eric Evans – US FDA, Center for Veterinary Medicine, USA
O-8 **Liquid Chromatography-Tandem Mass Spectrometry for the Simultaneous Determination of Multiple Hormones in Fish Muscle**

3:55 – 4:20 pm **Bruno Giri**, Lynn Cruickshank – USDA Food Safety Inspection Service, Eastern Laboratory, USA
O-9 **Application of DNA Barcoding Techniques For Speciation in an FSIS Regulatory Laboratory**

4:20 – 4:45 pm **Richard Fussell**, Jonathan Tarbin, Danny Chan, Matthew Sharman – Food and Environmental Research Agency, UNITED KINGDOM
O-10 **Approaches to the Identification of Metabolites and Breakdown Products of Veterinary Drug Residues Using Accurate Mass Time-of-Flight Mass Spectrometry**

4:45 – 5:10 pm **Lucía Geis-Asteggiante**, Steven Lehotay, Alan Lightfield – USDA Agricultural Research Service, USA and Universidad de la República, URUGUAY
O-11 **Multiclass, Multiresidue Method (MMM) for More Veterinary Drug Residues in Animal Tissues**

6:00 pm Social Event: Dinner Cruise! TradeWinds Hotel Entrance
10:00 pm Return from Dinner Cruise Dock where Buses parked

Tuesday, July 17, 2012

7:15 – 8:15 am Early Morning Coffee Pavilion
7:30 – 4:30 pm Registration Grand Palm Colonnade
All Day Exhibition & Posters Pavilion

7:15 – 8:15 am **AB SCIEX Breakfast Seminar** **Horizon Rooms**
V-5 **Advanced Tools to Screen for Known and Unknown Chemical Residues in Food Samples**
André Schreiber, AB SCIEX, Concord, Ontario, CANADA

8:30 – 10:35 am **Global Trade Issues/International Monitoring** Ballrooms
Moderator: Johannes Corley, IR-4, Rutgers University

8:30 – 8:55 am **Carmen Tiu**, Kenneth Racke – Dow AgroSciences LLC, USA
O-12 **Pesticides Residues in Food and World Trade: Regulation and Monitoring**

8:55 – 9:20 am **Lucy Namu**, Kenya Plant Health Inspectorate Service (KEPHIS), Nairobi, KENYA
O-13 **Perspective from Africa** *(tentative)*

9:20 – 9:45 am **Árpád Ambrus**, Zsuzsanna Horváth, Sali Judit, Andrea Zentai – National Food Chain Safety Office, HUNGARY
O-14 **Principles for Planning Monitoring Pesticide Residues in Agricultural Commodities**

9:45 – 10:10 am **MeeKyung Kim**, Dong-Gyu Kim, Jin-Young Shin, Kyung Yuk Ko, Chae-mi Lim, Seong-Wan Son – Animal, Plant and Fisheries Quarantine and Inspection Agency, SOUTH KOREA
O-15 **The Korean National Residue Program for Veterinary Drugs, Pesticides, and Environmental Contaminants in Food of Animal Origin**

10:10 – 10:35 am Wei Wang, Jessica Boyd, Haiying Du, **Xing-Fang Li** – University of Alberta, CANADA
O-16 **Analytical Opportunities in Drinking Water Safety**

10:35 – noon **Exhibition & Posters** Pavilion

11:00 – noon **Poster Session B: Authors for EVEN number posters present**
Paul Yang and Lawrence Zintek co-coordinators of poster sessions

12:15 – 1:15 pm **UCT Lunch Seminar** **Horizon Rooms**
V-6 **Strong or Weak Ion Exchange: Their Impact on Extraction Outcomes**
Mike Telepchak and Brian Kinsella, UCT, Bristol, PA, USA

1:30 – 3:00 pm **Southern US State Laboratories Report Their Chemical Residue Activities: Doing More with Less** Ballrooms
Moderator: Sherry Garris, South Carolina Dept. of Agriculture
O-17

Sherry Garris, Chanin Craft – South Carolina Department of Agriculture
Perspective from South Carolina (Pyrethroids)

Jo Marie Cook – Florida Department of Agriculture and Consumer Services
Perspective from Florida (Seafood Analyses)

Loretta Fourrier – LSU Ag Chemistry Laboratory/LSU Ag Center;
Nina Huffstetler – Louisiana Department of Agriculture and Forestry;
Gale Hagood – Mississippi State Chemical Laboratory;
Joe Basile – Alabama Dept. of Agriculture & Industries
Perspective from Louisiana, Mississippi, and Alabama (PAHs)

Susan Eigen – Georgia Department of Agriculture
Perspective from Georgia (Antibiotics in Feed)

Input from Other States/Open Discussion

3:00 – 3:45 pm **BREAK** (Exhibition & Posters) Pavilion

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

First Half Focus: Targeted Analysis
Second Half Focus: Nontarget Analysis

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

7:00 – 8:00 pm Reception by TradeWinds Pavilion
8:00 pm → Night Beach Volleyball Game On the Beach

Wednesday, July 18, 2012

7:15 – 8:15 am Early Morning Coffee Pavilion
7:30 – noon Registration Grand Palm Colonnade
Until noon Exhibition & Posters Pavilion

7:15 – 8:15 am **Agilent Breakfast Seminar** **Horizon Rooms**
V-7 **Improving the Quality of your MS Data by Implementing Sample Preparation Techniques**
Joanie Stevens, Agilent Technologies, Inc.

and

Analysis of Known and Unknown Food Contaminants using a New GC/Q-TOF
Phil Wylie, Agilent Technologies, Inc.

8:30 – 10:35 am **Mega Methods and HRMS Data** Ballrooms
Moderator: Paul Yang, Ontario Ministry of the Environment

8:30 – 8:55 am **Walter Hammack** – Florida Dept of Agriculture and Consumer Services, USA
O-18 **Data Strategies for HRMS Food Analysis**

8:55 – 9:20 am **Jack Cochran**, Michelle Misselwitz, Julie Kowalski – Restek Corp., USA; Mark Merrick – LECO Corp., USA
O-19 **Comprehensive Two-Dimensional Gas Chromatography (GCxGC) for Mega Multi-Analyte Methods**

9:20 – 9:45 pm **Brian Eitzer**, Walter Krol – The Connecticut Agricultural Experiment Station; Walter Hammack – Florida Dept. of Agriculture; Michael Filigenzi – The California Animal and Food Safety Laboratory; Karen Wolnik, Lora Lin, Sara Robison – US FDA, Forensic Chemistry Center, USA
O-20 **The Use of High Resolution Liquid Chromatography/High Resolution Mass Spectrometry in Screening Produce for Pesticide Residues**

9:45 – 10:10 am **Kai Zhang**, Jon Wong, Paul Yang, Douglas Hayward, Alex Krynitsky – US FDA, Center for Food Safety and Applied Nutrition, USA and Ontario Ministry of the Environment, CANADA
O-21 **The Issues with Multiresidue Methods for Pesticide Analysis in Food**

10:10 – 10:35 am **Paul Yang**, Serei Thach – Ontario Ministry of the Environment, CANADA
O-22 **Analysis of Pharmaceuticals, Steroids and Antibiotics using UHPLC – Orbitrap Mass Spectrometry with Enhanced Sensitivity, Selectivity and Minimal Matrix Effects**

10:35 – noon **BREAK** (Exhibition & Posters) Pavilion

11:00 – noon **Posters Session C: Authors for ALL posters present**
Paul Yang and Lawrence Zintek co-coordinators of poster sessions

12:15 – 1:15 pm **BUCHI Lunch Seminar** **Horizon Rooms**
V-8 **Buchi SpeedExtractor – New Dimensions in Pressurized Solvent Extraction for Pesticides and Environmental Contaminants**
Susanne Feifel, Product Manager Extraction Solutions, and William Ickes, Product Specialist Extraction Solutions, BUCHI Corporation, New Castle, DE

1:30 – 2:45 pm **Residues in the Environment** Ballrooms
Moderator: Brad Barrett, AB Sciex

1:30 – 1:55 pm **Imma Ferrer**, E. Michael Thurman – University of Colorado, Center for Environmental Mass Spectrometry, USA
O-23 **LC/Q-TOF-MS for the Analysis of Emerging Contaminants in Water Samples**

1:55 – 2:20 pm David Wilson, Lorraine Iverson, **Margie St. Germain** – EPA Region 7, USA
O-24 **Rapid PCB Micro-Extraction of Soil Samples: Method Development**

2:20 – 2:45 pm **Sara Lupton**, Janice Huwe, Emilio Esteban, Otis Miller – USDA, Agricultural Research Service, Biosciences Research Laboratory and USDA FSIS, USA
O-25 **Dioxins and Other Contaminants in Catfish from U.S. Commerce**

2:45 – 3:00 pm **BREAK** Grand Palm Colonnade

3:00 – 4:40 pm **"Cream of the Crop Technical Talks"** Ballrooms
Moderator: Kate Mastovska, Covance Laboratories

3:00 – 3:20 pm **Lorraine Iverson**, Margie St. Germain – EPA, Region 7, USA
O-26 **Using Stir-Bar Sorptive Extraction (Twister™) for PAHs, Pesticides and PCPPs Analysis of Surface Water**

3:20 – 3:40 pm **John Budin** – Mérieux Nutrisciences Corp., USA; Donna Zink – AIM Research Enterprises LLC, USA
O-27 **Reference Material Database provided by the TDRM of AOAC INTERNATIONAL**

3:40 – 4:00 pm **Simon Hird**, George Stubbings – Food and Environmental Research Agency, UNITED KINGDOM
O-28 **The Challenges Faced During Development and Validation of Methodology Based on Liquid Chromatography–Tandem Mass Spectrometry (LC-MS/MS) for the Monitoring for Use of Anabolic Steroids in Animals**

4:00 – 4:20 pm **Robert Voyksner**, Sheng-Suan Cai, Jennifer Voyksner, Brian Nies, Jack Syage – LCMS Limited, USA and Syagen Technology, USA
O-29 **Use of APPI to Minimize ESI Matrix Effects in LC-MS Analysis of Pesticides in Foods**

4:20 – 4:40 pm **Yelena Sapozhnikova**, Stephen Timko, Edward Wirth, Michael Fulton, Steve Lehotay – USDA Agricultural Research Service, University of South Carolina, and Center for Coastal Environmental Health and Biomolecular Research, USA
O-30 **Analysis of Emerging Environmental Contaminants: Where We Are Now, and Thoughts About Where We Should Go**

4:40 – 5:00 pm **Closing** Ballrooms
Poster Awards and NACRW Logo Selection!

Meeting Adjourns

Mark your calendar for next year's workshop, July 21-24, 2013

Come celebrate with us!
FPRW/NACRW will be celebrating 50 YEARS
and our new name
North American Chemical Residue Workshop

POSTERS

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

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

Session C *All Authors stand by their posters from 11:00 am-noon on Wednesday*

P1 **A Compound-based Approach to Simplify GC-MS/MS Method Development for 280 Pesticides in Food Matrices**
Ed George et al. Bruker Daltonics Inc.- Chemical and Applied Markets, Fremont, CA, USA

P2 **Children's Food Project – Survey of Pesticide Residues in Infant Foods**
Angela Murray et al.; Canadian Food Inspection Agency, Ottawa, Ontario, CANADA

P3 **Automated extraction and sample cleanup for GC of Chlorinated Pesticides by EPA Method 8081**
Tom Hall and Jim Westmoreland, SCI-CON/FMS, Winter Park, FL, USA

P4 **Analysis of 136 Pesticides in Avocado using a Modified QuEChERS Method with LC-MS/MS and GC-MS/MS**
Narong Chamkasem, et al.; US FDA, SRL, Atlanta, GA, USA

P5 **Rapid Screening for Multiclass Pesticides in Meat at the USDA -Food Safety and Inspection Service (FSIS).**
Son Hoang et al.; USDA - FSIS - Office of Public Health Science - Western Laboratory, Alameda, CA, USA

P6 **Strategies for Building Broad Multi-Residue Screens by LC/GC-Mass Spectrometry**
Kelly Dorweiler et al.; General Mills/Medallion Laboratories Golden Valley, MN, USA

P7 **Standard Response Stability of Trace Pesticides in QuEChERS Cantaloupe Extracts: An Inlet Comparison**
Ryan C. Venable and Raymond A. Allum, Bureau of Chemical Residue Laboratories, Division of Food Safety, Florida Department of Agriculture and Consumer Services, Tallahassee, Florida, USA

P8 **Monitoring of pesticides residues in Paprika Using GC-MS/MS from Gyeongnam region in Korea**
Mi yeong Kim et al.; Gyeongnam Provincial Office, National Agricultural Products Quality Management Service (NAQS), SOUTH KOREA

P9 **Determination of Maleic Hydrazide Residue in Tobacco by LCMSMS and the QuEChERS**
Meng Yu et al.; Lancaster Laboratories, Inc. , Lancaster, PA, USA

P10 **Results for a Pesticide Monitoring Program of German Malts**
Mehmet Coelhan and Fritz Jacob, Technische Universität München, Research Center Weihenstephan for Brewing and Food Quality, GERMANY

P11 **Effects of Including or Removing of Sweet Corn Silk on the Levels of Pesticide Residues in Flesh Sweet Corn Samples**
Kazuaki Iijima et al.; The Institute of Environmental Toxicology (IET), JAPAN

P12 **The QuEChERS Sample Preparation Approach with Dispersive and Cartridge SPE Cleanup, GCxGC-TOFMS, and LC-MS/MS for the Analysis of Pesticides in Tobacco**
Michelle Misselwitz et al.; Restek Corporation, Bellefonte, PA, USA

P13 **Multi-Residue Pesticides Analysis in Herbal Tea Products by GC-MS/MS**
David Steiniger et al.; Thermo Fisher Scientific, Austin, TX, USA

P14 **Quantification of Pesticides Residues in Tomato Samples Using a Fast Multi-Residue Method by Liquid Chromatography Tandem Mass Spectrometry Combined with QuEChERS method**
Graziela C.R. Moura-Andrade et al.; Universidade de São Paulo, Centro de Energia Nuclear na Agricultura, BRASIL

P15 **Market Basket Survey of Organochlorine Pesticide Residues in Herbs and Spices Using EPA 1699**
Andrew Patterson et al.; Vista Analytical Laboratory, El Dorado Hills, CA, USA

P16 **Rapid Method for the Confirmatory Analysis of Paraquat and Diquat in Potatoes and Cereals**
Richard Fussell et al.; Food and Environment Research Agency, York, UK

P17 **Detection of Underivatized Glyphosate and Similar Polar Pesticides in Food of Plant Origin by LC-MS/MS**
Rolf Kern et al.; AB SCIEX, Foster City, CA, USA

P18 **Multiresidue Analysis of Rodenticides in a Food Matrix Using an Alkaline Extraction and Ultra Performance Liquid Chromatography/Tandem Mass Spectrometry**
Lynda Podhorniak and Yaorong Qian, U.S. EPA Analytical Chemistry Branch, Fort Meade, MD, USA

P19 **The Use of Microflow UHPLC as a Way to Reduce Solvent Usage in Pesticide Screening of Food Samples by LC-MS/MS**
Brent Lefebvre et al.; AB SCIEX, Concord, Ontario, Canada

P20 **A High-Throughput Approach to Multi-Matrix Food Testing Using QuEChERS and Tandem GC/MS**
Joan Stevens and Melissa Churley, Agilent Technologies, Lake Forest, CA, USA

P21 **Chromatographic Results for Several Structural Isomers Obtained from Sub 2μ vs. Conventional 3μ C18 Columns**
Ajayi Olusegun et al.; Pacific Regional Laboratory, Southwest, FDA, Irvine, CA, USA

P22 **QuEChERS in the Rye: Cleanup Comparison on QuEChERS extracts for Grains**
Jessica Netzer et al.; J2 Scientific, Columbia MO, USA

P23 **Maximizing Lipid Load With 2-Dimensional GPC Cleanup**
Jessica Netzer et al.; J2 Scientific,; Columbia MO, USA

P24 **Combining Dispersive Solid Phase Extraction Cleanup and Particulate Filtration for QuEChERS Extracts in One Simple Step**
Jack Cochran et al.; Restek Corporation, Bellefonte, PA, USA

P25 **Feasibility of Dilute-and-Shoot LC/MS/MS, Solvent-Only Calibration and Multiple Food Types for Multi-Residue Pesticide Analysis: Lazy Chemists and Old Instruments**
Julie Kowalski et al.; Restek Corporation, Bellefonte, PA, USA

P26 **Use of New Spherical Materials in a Dual-Layer SPE Tube for the Analysis of Pesticide Residues in Spinach**
Katherine K. Stenerson et al.; Sigma-Aldrich/Supelco, Bellefonte, PA, USA

P27 **Analysis of Pesticides in Fruits and Vegetables using Z-Sep and Z-Sep+ SPE Sorbents as Part of QuEChERS Approach to Sample Cleanup**
Katherine K. Stenerson et al.; Sigma-Aldrich/Supelco, Bellefonte, PA, USA

P28 **Pesticide Analysis Using QuEChERS Extraction: A Comparison of Manual and Automated Approaches**
Tyler Trent et al.; Teledyne Tekmar, Mason, Ohio, USA

P29 **Extraction of Spices using the SpeedExtractor E-916 for the Determination of Pesticides**
Feifel S et al.; Kantonales Labor Zurich, SWITZERLAND

P30 **Direct Sample Analysis: A New Ionization Technology for Rapid Pesticide Screening & Streamlined Analysis**
Courtney Phillips, et al.; PerkinElmer, Shelton, CT, USA

P31 **A Simple Method for Determination of Fungicides and Metabolites in Orange and Apple Juices using Polymeric SPE and LC/MS/MS**
Irina Dioumaeva, Agilent Technologies, Inc. Lake Forest, CA, USA

P32 **Multiresidue Method for Determination of Eight New Generation Fungicides IN Grapes Using GC- μECD**
Vilca.F.Z et al.; Center of Nuclear Energy in Agriculture CENA. University of Sao Paulo, Sao Paulo, BRASIL

P33 **Strategies for Rapid Screening and Quantitation of Pesticides by Direct Analysis in Real Time (DART) Ambient Ionization Mass Spectrometry**
Elizabeth Crawford and Brian Musselman, IonSense, Inc., Saugus, MA, USA

P34 **A Rapid Response for the High Throughput, Sensitive and Accurate Determination of Carbendazim from Juice and Juice Concentrate**
Frederic Ciner and Ed Dabrea, Jupiter Analytix/Jupiter Environmental Labs, Jupiter, FL USA

P35 **Determination of Carbendazim in Orange Juice by QuEChERS and LC/MS/MS Detection**
Xiaoyan Wang and Michael J. Telepchak, UCT, Bristol, PA, USA

P36 **Speciated Arsenic Contents of Some Foods**
John Budin et al.; Mérieux NutriSciences Corporation, Chicago, IL USA

P37 **Rapid Detection of Pesticides in Fruit Juice Without Sample Preparation Using High Resolution Chromatography and Highly Sensitive Tandem MS**
Ken Rosnack et al.; Waters Corporation, Milford, MA, USA

P38 **Fungicides in Fruit Juice: Sample Preparation and Analytical Strategies for Multiresidue Screening, Targeted, and Confirmatory Analysis**
Michael S. Young and Kim vanTran, Waters Corporation, Milford, MA, USA

P39 **Inter-laboratory Validation of a Fast and Sensitive UHPLC-MS/MS Method with Fast Polarity Switching for the Analysis of Lipophilic Shellfish Toxins**
Jerry Zweigenbaum et al.; Agilent Technologies, Wilmington, DE, USA

P40 **Development and Validation of a Method for Stilbene Residues in Fish and Fish Products**
Lynn Watson et al; Canadian Food Inspection Agency, Dartmouth, NS, CANADA

P41 **Simultaneous determination of antibiotic residues in water from fish farm cage net by LC-MS/MS**
Sérgio H. Monteiro et al.; Universidade de São Paulo, Centro de Energia Nuclear na Agricultura, BRASIL

P42 **Imported Catfish and Food Safety**
Cindy Foster, et al.; Mississippi State Chemical Laboratory, Mississippi State, MS, USA

P43 **Detection of the Illegal Use of Trifluralin in Shrimp Farming**
Richard Fussell et al.; Food and Environment Research Agency, Sand Hutton, York, UK

P44 **Decreasing the Impact of Aflatoxin in Mississippi**
Ashli Brown et al.; Mississippi State University, Department of Biochemistry, Molecular Biology, Entomology, and Plant Pathology, Mississippi State, MS, USA

P45 **Improved Method for Determination of 3-chloro-p-toluidine hydrochloride (DRC-1339) Residues in Avian Tissues by GC/MS/MS**
Steven F. Volker and Randal S. Stahl, National Wildlife Research Center, Fort Collins, CO, USA

P46 **Analysis of Stilbene Residues in Aqucultured Finfish using LC-MS/MS**
Wendy C. Andersen et al.; US Food and Drug Administration, Animal Drugs Research Center, Denver, CO, USA

P47 **Sample Preparation for UPLC-MS/MS Determination of Aminoglycoside Antibiotics in Meat and Milk**
Michael S. Young et al.; Waters Corporation, Milford, MA; USA

P48 **Multi-Target Comparative Screening for Pesticides using High Resolution and Accurate Mass LC-MS/MS**
Andre Schreiber and Axel Besa, AB SCIEX, Concord, Ontario, CANADA

P49 **Identification of Pesticide Residues in Food Samples using LC-MS/MS and Non-Targeted Data Processing Strategies**
Scott Kragerud, et al.; AB SCIEX, Foster City, CA, USA

P50 **Screening of Unknown Pollutants in Water Sample Using High Resolution and Accurate Mass LC-MS/MS**
YunYun Zou, et al.; AB SCIEX, Concord, Ontario, CANADA

P51 **Finding Pesticides using an Agilent 7200 GC/Q-TOF and Unknowns Analysis Software**
Philip L. Wylie, Agilent Technologies, Wilmington, DE, USA

P52 **Ultra-high Performance Liquid Chromatography Coupled with Electrospray Ionization Quadrupole Time-of-Flight Mass Spectrometry for Simultaneous Screening of Multiple Pesticides in Scallion**
Shan Zhou et al.; WW Food Team China Segment, Chemical Analysis Group, Agilent Technologies, Beijing, CHINA

P53 **Analysis of Multiple Pesticide Residues in Scallion by Gas Chromatography Quadrupole Time of Flight–Mass Spectrometry (GC /Q-TOF)**
Wenwen Wang and Shan Zhou, Agilent Technologies, Beijing, CHINA

P54 **Exact Mass MS/MS Spectral Library for Screening Non-targeted Pesticides and Other Contaminants in Food and Environmental Samples using LC-QTOF-MS**
Jerry Zweigenbaum et al,; Agilent Technologies, Wilmington, DE USA

P55 **Differential Analysis of Organic and Non-Organic Honey for Pesticides and Pollutants by LC Time-of-Flight Mass Spectrometry**
Joan Stevens et al.; Agilent Technologies, Lake Forest, CA, USA

P56 **Novel Scientific Information System Evaluated for Semi-Quantitative High Throughput Screening of Pesticide Residues in Food**
Ken Rosnack et al.; Waters Corporation, Milford, MA, USA

P57 **Utilization of Rapid LC-MS for Screening and Quantitative Analysis of Pesticides in Food Matrix using an Orbitrap Platform**
Michal Hauer et al.; Thermo Fisher Scientific, West Palm Beach, FL, USA

P58 **Multi-residue Pesticide Analysis of Botanical Dietary Supplements using Gas Chromatography- Tandem Mass Spectrometry (GC-MS/MS) and GC-High Resolution-Time-of-Flight (GC-HRT)**
Douglas G. Hayward et al.; U.S. Food and Drug Administration, Center for Food Safety and Applied Nutrition, College Park, MD, USA

P59 **Quick and Easy Screening and Quantitation of Pesticides in Food and Biological Matricies using Orbitrap Mass Spectrometer**
Marcus Miller et al.; ThermoFisher Scientific, San Jose, CA, USA

P60 **Simultaneous Determination of Paraquat and Diquat in Environmental Water Samples by HPLC-MS/MS**
Marcus Miller et al.; ThermoFisher Scientific, San Jose, CA, USA

P61 **High Resolution LC-MS for Screening and Quantitative Analysis of Antibiotics in Drinking Water using an Orbitrap and Online Sample Preparation**
Cory Lytle et al.; Thermo Fisher Scientific, West Palm Beach, FL, USA

P62 **Use of D6 Internal Standards for NCI-GC-MS Analysis of Eight Pyrethroids in Environmental Samples**
Del A. Koch, ABC Laboratories, Columbia, MO, USA; and Kevin Clark, Morse Laboratories, LLC, Sacramento, CA, USA

P63 **Insecticide Usage Pattern on Brinjal and Resistance in Brinjal Shoot and Fruit Borer (BSFB) and Residue in Brinjal**
Dr. Md Arifur Rahman, Entomology Division, Bangladesh Agricultural Research Institute, Gazipur, BANGLADESH

P64 **Development of a GC-MS/MS Based MRM Method for Quantitation of PCB and PBDE Congeners in Human Plasma**
Helen (Qingyu) Sun et al.; Bruker Daltonics, Chemical Applied Markets Division, Fremont, California, USA

P65 **Determination of 4-Nonylphenol in Biosolids and Biosolids-Applied Soil Using SpeedExtractor and GC/MS/MS**
William H. Ickes, et al.; Buchi Corporation, New Castle, DE, USA

P66 **Use of Banana Peel for Removing Atrazine and Ametryne from Natural Waters**
Sérgio H. Monteiro et al.; Center for Nuclear Energy in Agriculture, University of Sao Paulo, BRASIL

P67 **Development of QuEChERS/Low-Pressure Fast GC-MS/MS Method for the Analysis of Tris(2-Chloroethyl) Phosphate in Rice**
Natchanun Leepipatpiboon et al.; Chromatography and Separation Research Unit, Department of Chemistry, Faculty of Science, Chulalongkorn University, Bangkok, THAILAND

P68 **Advances in Automating Solid Phase Extraction for UCMR3: Hormones and 1, 4-Dioxane**
Michael Ebitson and David Gallagher, Horizon Technology Inc., Salem, NH, USA

P69 **Mitigation of Off-Site Mobility of Dimethazone and Trifluralin Herbicides**
George F. Antonious, College of Agriculture, Food Science, and Sustainable Systems, Division of Environmental Studies, Kentucky State University, Frankfort, KY, USA

P70 **Comprehensive Analysis of Persistent Organic Pollutants Using High Performance Time-of-Flight Mass Spectrometry**
David E. Alonso and Joe Binkley, LECO Corporation, St. Joseph, MI, USA

P71 **Evaluation of Measuring Pyrethroids in Sediment Using Solid Phase Extraction and GC/MS-NCI and GC/MS/MS-EI**
Jona Verreth and Angela Schaner, Montana Department of Agriculture, Bozeman, MT, USA

P72 **Efficient Pesticides Remediation using OPH enzyme bearing Metal Organic Framework (MOF)**
Yongwoo Lee et al.; QinetiQ North America, Inc., Waltham, MA, USA

P73 **Lowering Detection Limits for 1,4-Dioxane in Drinking Water Using Concurrent Solvent Recondensation - Large Volume Splitless Injection in an Unmodified Split/Splitless GC Inlet**
Jack Cochran et al.; Restek Corporation, Bellefonte, PA, USA

P74 **The Holy Grail: Comprehensive Polyaromatic Hydrocarbon Analysis by Serial Combination of HPLC Columns with Different Selectivities and UV and Fluorescence Detection**
Julie Kowalski et al.; Restek Corporation, Bellefonte, PA, USA

P75 **Characterization of Crude Oils and Tar Balls using Biomarkers and Comprehensive Two-Dimensional Gas Chromatography**
Michelle Misselwitz et al.; Restek Corporation, Bellefonte, PA, USA

P76 **Determination of PAHs in Fish by QuEChERS Extraction and Dual Layer SPE Cleanup**
Xiaoyan Wang and Michael J. Telepchak, UCT, Bristol, PA, USA

P77 **Estimation of measurement uncertainty for determination of pesticide residues in soil by liquid chromatography-mass spectrometry**
Diego. A. Ahumada F et al.; Laboratorio de Inocuidad Química. Centro de Bio-Sistemas. Universidad de Bogotá, Bogota COLUMBIA

P78 **Sorption and desorption of ametryn and glyphosate applied in association in soil of sugarcane cultivation: Could a molecule interfere on the behavior of the other in association?**
Paulo A. de Toledo Alves et al.; Center for Nuclear Energy in Agriculture CENA/USP, Piracicaba SP, BRASIL

P79 **Reduction of Mancozeb Residues on Tomatoes by Dipping Solutions**
Mehmet F. Cengiz and Muharrem Certel, Akdeniz University, Faculty of Engineering, Food Engineering Department, Antalya, TURKEY

P80 **Determination Of Organochlorine Pesticides In Sea Turtle Liver Using A Multiresidue Method and GC-µecd**
Angélica María Sánchez-Sarmiento et al.; Department of Pathology, College of Veterinary Medicine and Zoothechny, University of São Paulo, São Paulo, BRASIL

P81 **Monitoring Of Organochlorine Pesticides In Green Sea Turtle Fat Using A Multiresidue Approach Method and GC-µecd**
Angélica María Sánchez-Sarmiento et al.; Department of Pathology, College of Veterinary Medicine and Zoothechny, University of São Paulo, São Paulo, BRASIL

P82 **Polybrominated Diphenyl Ethers (PBDEs) in Surface Sediments of Hillsborough Bay, FL**
Candice Simmons and Foday Jaward, **College of Marine Science**, University of South Florida, St. Petersburg, FL, USA

P83 **Fate of Imidacloprid and its Metabolites in Honey Bee Hives**
Alaa Kamel, et al.; Analytical Chemistry Laboratory, Biological and Economic Analysis Division, Office of Pesticides Program, US EPA, Fort George G. Meade, MD, USA

P84 **Benefits and Limits of an LC-MS/MS Based Online Sample Clean-up Method for High Throughput Analysis of Pesticide Residues in Different Food Matrices with Regards to the Latest Carbendazim Contamination Issue in Orange Juice**
Laszlo Hollosi et al.; Food Safety Response Center, Thermo Fisher Scientific, Steingrund , Dreieich, GERMANY

P85 **A New Sample Preparation Method for Malachite Green and other Food Dyes in Salmon by LC-MS-MS**
Elena Gairloch and Mats Leeman, Biotage, Charlotte, NC, USA

P86 **Monitoring of Pesticide Residue in Agricultural Products using GC/MS/MS**
Nam Suk Kang et al.; Busan Regional Korea Food and Drug Administration, Busan, KOREA

P87 **High Throughput Quantitative Analysis of Pesticides by Gas Chromatography with Triple Quadrupole Mass Spectrometry**
Ryan Picou and John Reuther, Eurofins Analytical Labs, Metairie, LA, USA

P88 **Internship Program - Unique Opportunity for Future Scientists**
Yelena Karaseva, U.S. FDA, Northeast Regional Laboratory, Jamaica, NY, USA

P89 **An automated, chronic, pesticide bio-assay, based upon reductions in *Vibrio fischeri* luminescence, can be used to screen for the presence of Dibrom toxicity in natural waters, with an EC_{50} of ~0.4ppm**
Jose Barreto, et al.; Florida Gulf Coast University (FGCU) Green Technology Research Group and Department of Chemistry, South Fort Myers, FL, USA

P90 **Automated Extraction and Analysis of Mycotoxins from Food Products Using DPX-LC/MS/MS**
Oscar Cabrices et al.; GERSTEL Inc., Linthicum, MD, USA

P91 **Monitoring of Hexaconazole Residues in Agricultural Products of Korea**
In-Kyung Kim et al.; Gwangju Regional Korea Food & Drug Administration, Gwangju, KOREA

P92 **A Rapid Method for the Determination of Multi-Mycotoxins in Wines and Beer by LC-MS/MS using a Stable Isotope Dilution Assay**
Fadwa Al-Taher, et al.; Institute for Food Safety and Health, Bedford Pk, IL, USA

P93 **Determination of Antibiotic Residues in Milk and Dairy Products**
Tom Dobbs et al.; J2 Scientific, Columbia MO USA

P94 **Selective and Sensitive Analysis of Organophosphorus Pesticides in Baby Food Using an Inexpensive GC Detector**
Scott Hazard and Gary Engelhart, OI Analytical, College Station, TX, USA

P95 **Analysis of Antioxidants in foods and Dietary Supplements Using HPLC with Post-Column Derivatization**
Wendy Rasmussen, and Maria Ofitserova, Pickering Laboratories, Inc. Mountain View, California, USA

P96 **High Throughput LC-MS-MS Analysis of Carbendazim in Orange Juice**
Jeffrey H. Dahl and William Hedgepeth, Shimadzu Scientific Instruments, Columbia, MD, USA

P97 **Increasing Speed of UHPLC-MS Analysis using Single-Stage Orbitrap Mass Spectrometer**
Maciej Bromirski and Olaf Scheibner, Thermo Fisher Scientific, Bremen, GERMANY

P98 **Detection and Quantitation of Brominated and Chlorinated Hydrocarbons by DART with Linear Ion Trap and Triple Quadrupole Technology**
Michal Hauer, et al.; Thermo Fisher Scientific, West Palm Beach, FL, USA

P99 **Evaluating the Effectiveness of Incorporating Ceramic Homogenizers in the QuEChERS Extraction Method**
Brian Kinsella et al.; UCT Inc., Bristol, PA 19007, USA

P100 **A Field Study on the Removal and Destruction of Polychlorinated Biphenyls from Vertical Painted Surfaces**
Simone E. Novaes-Card et al.; University of Central Florida, Orlando, FL, USA

P101 **Development of *In situ* Degradation Technologies for Chlordane Residues**
Tamra Legron-Rodriguez et al.; University of Central Florida, Orlando, FL, USA

P102 **Self-Carmelization of Sucralose: Identification of over 100 Degradation Products by UHPLC and LC/QTOF-MS with Ultrahigh Resolution time-of-flight mass spectrometry and Accurate Mass**
E. Michael Thurmam et al.; University of Colorado, Department of Environmental Engineering, University of Colorado, Boulder, CO USA

P103 **Automated QuEChERS Tips for GC/MS Analysis of Fruit and Vegetables**
William E. Brewer et al.; Dept. of Chemistry and Biochemistry, University of South Carolina, Columbia, SC, USA

ORAL PRESENTATION ABSTRACTS

O-1 Pesticide Residue Testing Requirements under the National Organic Program

Gwendolyn Wyard, **Organic Trade Association**, 28 Vernon St; Brattleboro, VT 05301, USA; gwyard@ota.com; www.ota.com

Under the Organic Foods Production Act (OFPA) of 1990, the National Organic Program (NOP) is authorized to implement regulations that require accredited certifying agents to conduct residue testing of organically produced agricultural products. The main objectives of the residue testing program are to: (1) ensure that certified organic production and handling operations are in compliance with the requirements set forth in NOP regulations; and (2) serve as a means for monitoring drift and unavoidable residue contamination of agricultural products to be sold or labeled as organically produced. Chemical analysis must be made in accordance with the methods described in the most current edition of the *Official Methods of Analysis of the AOAC International* or other current applicable validated methodology. NOP recently issued a Proposed Rule clarifying that in accordance with OFPA, certifying agents must regularly conduct residue testing, in addition to residue testing when there is reason to suspect contamination with a prohibited substance. NOP has also provided instructions on laboratory selection criteria and provided a list of target pesticides that are suggested to test for. When residue testing detects residues at levels greater than 5 percent of EPA's tolerance for the specific residue detected, the product must not be sold as organically produced. Pesticide residue test results that exceed FDA or EPA's regulatory tolerances must be promptly reported to the Federal health agency whose regulatory tolerance or action level has been exceeded. In addition to required testing by certifying agents, many certified operators voluntarily conduct residue testing.

O-2 Untargeted Residue Analysis and the "omics": The Study Of Health Impacts Of Residue Exposure - A Metabolomics Focus

Bruce Peebles[1,2], Catherine Rawlinson[1,2,] Joel Gummer[1,2], Garth Maker[1,2,3], Ian Mullaney[2,4], Robert Trengove[1,2]

[1]Murdoch University Metabolomics Australia Node, Murdoch University, 90 South Street, Murdoch WA 6150, Australia; [2]Separation Science and Metabolomics Laboratory, Murdoch University, 90 South Street, Murdoch WA 6150, Australia; [3]School of Biological Sciences and Biotechnology, Murdoch University, 90 South Street, Murdoch WA 6150, Australia; [4]School of Veterinary and Biomedical Sciences, Murdoch University, 90 South Street, Murdoch WA 6150, Australia; r.trengove@murdoch.edu.au

The "omics", particularly proteomics and more recently metabolomics have been drivers in recent instrumental development, leading to faster chromatography, accurate mass and high resolution mass analyzers, and equally as important robust sample interfaces for minimal sample preparation. Alongside the hardware developments there have been considerable developments in software development for mass spectral deconvolution, data visualisation, and mapping of analytes onto biochemical pathways to identify which pathways are modifed in a challenged sample vs a control sample. For instance, this allows the mechanisms of disease progression to be dissected and potential management strategies and potential therapeutic targets to be identified.

These same instrumental developments have been utilised by the residue and environmental communities with the development of multiresidue methods that screen for in excess of 1000 analytes in a single analysis. Metabolomics seeks to identify changes in response to some stimulus, whilst residue and environmental analysis seeks to identify all possible residue and environmental "stimulus".

We are at the emergence of the capability to simultaneously measure residue and environmental analytes and profiles of primary and secondary metabolites. We can start to investigate from both directions or investigate "cause" and "effect" simultaneously.

This presentation will address the approach and the challenges that need to be addressed to facilitate the use of "untargeted methods". In particular the different approaches required for GC/MS based vs LC/MS based metabolomics. Instrumentation optimisation will be discussed in the context of combination of targeted compound analysis and metabolite profiling.

Collaborative pathways to advance this approach will be discussed and the approach needed to expand this to other omics such as lipidomics and proteomics.

O-3 Arsenic Speciation in Fruit Juice and Rice Products Using HPLC-ICP-MS

Douglas T. Heitkemper[1], Nohora P. Shockey[1], Kevin M. Kubachka[1], Traci A. Hanley[1], and Sean D. Conklin[2]

[1]**FDA/ORA, Forensic Chemistry Center,** 6751 Steger Dr. Cincinnati, OH 45237, USA; douglas.heitkemper@fda.hhs.gov; [2]FDA/CFSAN, Chemical Contaminants Branch, College Park, MD

The US FDA routinely monitors food for total arsenic as part of the Total Diet Study and through a Toxic Elements Program. In response to recent reports from consumer groups and extensive media coverage, there has been increased interest in measuring individual arsenic species with an emphasis on inorganic arsenic in foods including fruit juice and rice products. Inorganic arsenic species are known to be more toxic than organically bound arsenic species. Arsenic speciation utilizes HPLC coupled to ICP-MS for the separation and element-specific detection of arsenic species including arsenite, arsenate, dimethylarsinic acid, and monomethylarsonic acid. HPLC-ICP-MS methods are capable of measuring low µg/kg levels of arsenic species in a variety of foods. A description of the methods currently in use along with figures of merit and some typical results for sample types will be presented. In addition, an overview of the various FDA activities surrounding arsenic speciation in foods will be discussed.

O-4 2011 Imprelis (Aminocyclopyrachlor) Crisis: What happened, how we dealt with the situation, and lessons learned.

[1]Ping Wan, [2]Steven C. Moser

[1]**Office of Indiana State Chemist**, 175 S. University Street; West Lafayette, IN 47907, USA; wanp@purdue.edu;
[2]**Oklahoma Dept. of Agriculture, Food, & Forestry**, 2800 N. Lincoln Blvd; OKC, OK 73105, USA; steven.moser@ag.ok.gov;

DuPont started selling Imprelis Herbicide in January 2011. In early spring of 2011 EPA Region III and V states started seeing tree damage and Imprelis was the suspected herbicide. Office of Indiana State Chemist was soon overwhelmed by suspected cases of Imprelis damage to trees. Indiana quickly became (not by choice) the hotspot for suspected Imprelis damage. The Laboratory would soon see over 400 samples pulled by field inspectors. In July, Indiana asked Oklahoma if they would be willing to look at the method and to check and see if there was a quicker/easier way to extract and analyze Imprelis. On August 11, 2011, the U.S. Environmental Protection Agency issued an order to DuPont directing the company to immediately halt the sale, use or distribution of Imprelis. This is the story of how the new herbicide Imprelis became a multistate issue for EPA FIFRA Laboratories, how Indiana and Oklahoma were involved, and what the two states experienced during and after the crisis.

O-5 Ion mobility-mass spectrometry for the screening of pesticides in food

Séverine Goscinny,[1] Laure Joly,[2] Edwin De Pauw,[2] Vincent Hanot,[1] and Gauthier Eppe;[2]

[1]**Scientific Institute of Public Health**, 14, rue Juliette Wytsman; 1050 Brussels, Belgium; sgoscinny@wiv-isp.be;
[2] University of Liège-Mass Spectrometry Laboratory; Liège, Belgium;

Nowadays, laboratories involved in the analysis of pesticide residues in food are facing a serious challenge: targeting an increase number of compounds at residue levels in complex matrices. A very attractive strategy to overcome this issue is the use of screening methods in conjunction with the feature of full scan mode of the time-of-flight analysers. This type of mass spectrometer solves only one facet of the challenge: the number of target compounds per acquisition. However, the issue of the low concentrations of the pesticides remains full when your extract has yield large amounts of co-extracted matrix compounds and contamination from solvents and reagents. In this case the identification process is very difficult and can taint the entire strategy.
We propose the use of Ion mobility (IM) coupled to mass spectrometry (MS) to overcome this problem. IM separates ions on the basis of their size/charge ratios and their interaction with a buffer gas. We will demonstrate how IM can resolve the target ions from the matrix interferences consequently improving the signal-to-noise ratio of characteristic ions.

Ion mobility mass spectrometry experiments were performed on a Synapt G2 spectrometer (Waters, Manchester, UK). The instrument will monitor the time required for an ion to travel through the mobility cell: the drift time. Spiked and previous EU proficiency tests samples were analyzed with an UPLC hyphenated to the Synapt G2. Based on these results, the presentation will show how the drift time can be used as a new criterion for identification purpose.

O-6 US EPA UPLC/MS/MS Method Development For Analytes Of Concern

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

The US EPA Chicago Regional Laboratory is developing methods for qualitative and quantitative determination for alkylphenols, alkylphenol ethoxylates and Bisphenol A in sludge, biosolids and soil for a Regional focused research effort on land application of biosolids and emerging contaminants. The development of ASTM Standards for hormones, Triclosan and other analytes of interest at the ppb to sub-ppt concentration levels in water and wastewater will be discussed. These methods utilize SPE extraction followed by UPLC/MS/MS analysis to achieve sub-ppt quantitation levels and the use of more sensitive mass spectrometers. Recently proposed, preliminary and approved ASTM Standards will be presented to demonstrate the process and relevance of the work. Laboratories interested in volunteering in multi-lab validations of ASTM Standards are encouraged to attend.

O-7 The Detection of Pesticides and Dioxins in Oils using Laser-Induced Breakdown Spectroscopy (LIBS)

Rosalie A. Multari,[1] David A. Cremers,[1] Thomas Scott,[2] and Peter Kendrick[2]

[1]**Applied Research Associates, Inc.**, 4300 San Mateo Blvd NE, Suite A-220, Albuquerque, NM, 87110, USA; rmultari@ara.com; [2]Diversified Laboratories; 4150 Lafayette Center Drive, Chantilly, VA 20151

The ability to detect and differentiate organic targets using laser-induced breakdown spectroscopy (LIBS) has become of increasing interest in recent years. LIBS is a particularly attractive analysis technique such applications because of its simplicity. A series of powerful laser pulses are directed at the target to form microplasmas. The plasma light is collected and spectrally analyzed to identify the target material. Analysis results are presented rapidly, within seconds to minutes. Advances in LIBS spectral data analysis using multivariate regression techniques applied to the entire LIBS spectrum have led to the ability to detect organic chemicals in complex matrices such as foods and on food processing equipment. Here, proof-of-principle data demonstrating the use of LIBS to detect Aldrin, 1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin, Chlorpyrifos, and Dieldrin in oils will be presented. The pesticide concentrations in the oils ranged from 0.005 μg/g to 0.1 μg/g. It was found that all samples could be successfully differentiated from each other by pesticide type and concentration. The samples could also be differentiated from control samples for the pesticide solvent and base oil.

O-8 Liquid Chromatography-Tandem Mass Spectrometry for the Simultaneous Determination of Multiple Hormones in Fish Muscle

Pak-Sin Chu, Wei Song, Tricia Johnson, Hiranthi Jayasuriya, and Eric Evans

U.S. Food and Drug Administration, Center for Veterinary Medicine, Office of Research, 8401 Muirkirk Road, Laurel, MD 20708, USA; pak.chu@fda.hhs.gov

In veterinary medicine, hormones are used to improve meat quality and feed efficiency in food-producing animals. Illegal use of hormones is of concern and may lead to harmful residues in edible tissues destined for human consumption. With the growing number of seafood products imported into the United States, specific and accurate assays are needed to measure multiple hormone residues in seafood products. We have developed a liquid chromatography-mass spectrometry/mass spectrometry (LC-MS/MS) method for quantitating multiple hormones in fish muscle. Analytes are extracted from fish tissue followed by sample clean-up and LC-MS/MS analysis. The sample cleanup involves a solvent partition and two solid-phase extractions. An atmospheric pressure chemical ionization (APCI) probe coupled with MS analysis in a rapid polarity switching mode is used to quantitate both positively and negatively charged hormones. For control muscle fortified at 0.5, 1, and 2 parts-per-billion, the method accuracies are >60% and CVs <20%. The method accuracy of 17β-estradiol is slightly higher than 110% at low fortification levels. Such effect may be attributed to the presence of trace level of endogenous 17β-estradiol in the control tissue. Sixteen hormones, including testosterone, cis-testosterone, progesterone, 19β-nortestosterone, 17α-methyltestosterone, trenbolone, melengestrol acetate, megestrol, 17β-estradiol, 17α-estradiol, ethynyl estradiol, zeranol, estriol, dienestrol, diethylstilbestrol, and hexestrol at 1 part-per-billion level can be determined in a single assay.

O-9 Application of DNA Barcoding Techniques For Speciation in an FSIS Regulatory Laboratory

Bruno J. Giri[1], Lynn C. Cruickshank[1]

[1]**USDA Food Safety and Inspection Service, Eastern Laboratory**, Richard B. Russell Research Center, 950 College Station Rd, Athens, GA, USA; bruno.giri@fsis.usda.gov

As part of the U.S. Farm Bill of 2008, regulatory oversight of the catfish industry was transferred to the USDA/FSIS. An important aspect of this regulation involved the establishment of species identity. To this end, the Eastern Laboratory's (EL) Chemistry Branch began working to adopt and verify use of appropriate methodology. The EL Chemistry screen for speciation employs water soluble protein electrophoresis (Isoelectric focusing or IEF) and confirmation with PCR/ DNA barcoding. DNA barcoding involves the amplification and sequencing of the mitochondrial cytochrome oxidase I (COI) gene. The COI gene exhibits a relatively fast mutation rate which leads to a significant variation between species while maintaining a relatively small variance within a species. Researchers have successfully barcoded more than 100,000 species of animals. Verification of the PCR method was recently completed. Verification data included collection of COI barcode sequences for approximately twenty authenticated specimens of both catfishes and non-catfishes. Future plans include the potential application of DNA barcoding to single animal species products of a wider variety including raw and cooked tissues. This would necessitate more extensive studies to show the applicability in such a diverse set of matrices.

O-10 Approaches to the Identification of Metabolites and Breakdown Products of Veterinary Drug Residues Using Accurate Mass Time-of-Flight Mass spectrometry

Richard J Fussell, Jonathan Tarbin, Danny Chan, and Matthew Sharman

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

The ability to detect and identify metabolites and breakdown products is important in the control of the unauthorised use of veterinary medicines, especially in cases where complex MRL definitions apply and the defined marker compounds cannot be included into screening methods. This presentation will describe an approach based on generic extraction and clean-up followed by LC-(q)ToF-MS with proprietary profiling and metabolite identification software packages to interrogate the raw data. Agilent MassHunter Qualitative software and Mass Profiler Professional (MPP) are used to identify statistically significant differences between sample extracts from treated and control animals. Also, Metabolite ID (MetID) software is used to scan raw data files for exact masses corresponding to predicted metabolites. The outputs from MPP and MetID are then applied to reverse search the raw data files using MassHunter Qualitative and Quantitative software, to confirm presence in dosed, and absence in control, samples, and provide theoretical empirical formulae. Re-analysis by LC-qToF-MS provides fragmentation/structural information and hence tentative identification which can be verified by comparison with the analytical standard, if available. If the analyte identified is a 'suitable marker of use', it can be incorporated into a monitoring programme. Examples will be used to illustrate this approach in greater detail.

O-11 Multiclass, Multiresidue Method (MMM) for more Veterinary Drug Residues in Animal Tissues

Lucía Geis-Asteggiante,[1,2] Steven J. Lehotay,[1] and Alan R. Lightfield[1]

[1] **USDA Agricultural Research Service**; 600 East Mermaid Lane; Wyndmoor, PA 19038; USA. lucia.geis@ars.usda.gov
[2] Cátedra de Farmacognosia y Productos Naturales, DQO; Facultad de Química; UdelaR; General Flores 2124; 12800, Montevideo; Uruguay

At FPRW last year, a practical and reliable MMM was developed by our research group for the analysis of 62 veterinary drug residues by UHPLC-MS/MS in bovine kidney. Based in this method, an extension to 128 drug analytes was optimized and validated in meat and other tissues. As instrument performance is of key importance for routine analysis, the optimization study centered on extensive ruggedness evaluation. Different clean-up sorbents were tested and amount of co-extractives calculated, matrix effects were evaluated using post-column infusion of representative analytes, the effect of dilution before injecting was studied, and recoveries and reproducibility were determined. Moreover, as the number of analytes increased, the wide range of chemical properties required the evaluation of different mobile phases and types of C-18. Ultimately, endcapped C-18 was chosen for cleanup and post-column infusion of ammonium formate was used as an ionization enhancer for the late-eluting anthelmintics. Considering all of these factors, a multi-day, multi-analyst validation was done using the final method, which obtained good qualitative performance and demonstrated that the method is suitable for screening purposes at or below US regulatory tolerance levels.

O-12 Pesticides Residues in Food and World Trade: Regulation and Monitoring

Carmen Tiu, Kenneth Racke

Dow AgroSciences LLC, 9330 Zionsville Road; Indianapolis, IN 46268, USA; tcarmen@dow.com

Within the current food-chain dilemma, the compromise has been set such that pesticides are authorized for use in the minimum amount to provide effective pest control and if residues are not harmful to human health. It is therefore imperative that residues are measured (by registrants) prior to authorizing use and setting tolerances (Maximum Residue Limit, MRL) that can be later monitored for compliance in food commodities (by governmental or private labs). In addition, there are secondary requirements set from the food-chain perspective to set arbitrary residue limits, typically lower than the established tolerances/MRL's. As per the United Nations' Food and Agriculture Organization (UN-FAO) 2010 statistics mention that the total ag-production is driven by cereal grains, oilseeds and meat commodities. However they are well covered by tolerances/MRL's, they are not harmonized between countries, which generates trade "irritants". There is also a large number of fruits and vegetable, particularly the one known as "minor crops" that lack tolerances/MRL's. The monitoring results indicate that import violations of residue levels may occur with low frequency (<1%) and are primarily a trade barrier, and usually not a health one. There are many national and international initiatives driven to establish more tolerances/MRL's and to harmonize existing ones, as well as to generate global packages supporting globally harmonized tolerances/MRL's. There are also initiatives to generate global MRL-databases and make them more readily available to farmers, training programs to avoid off-label uses and proper implementation of Integrated Pest Management (IPM) and proactive upfront checking of potential trade irritants.

O-14 Principles for planning monitoring pesticide residues in agricultural commodities

Árpád Ambrus, Zsuzsanna Horváth, Sali Judit, and Andrea Zentai

National Food Chain Safety Office, Directorate for Food Safety Risk Assessment, Tábornok u.2 H-1143 Budapest, Hungary; ambrusadr@yahoo.co.uk

Pesticide residues are monitored in very large number of samples compared to those analyzed in supervised trials to verify that the maximum residue limits (MRLs) properly cover residues deriving from pesticide applications according to registered use patterns under practical conditions, or whether the farmers follow the use recommendations. In addition, targeted sampling programs are conducted to control compliance with MRLs in locally marketed, imported and exported products.

The variability of average pesticide residues in lots of raw agricultural commodities is characterized with relative standard deviations of 50-100%. The variability of residues in crop units (single orange or head of cabbage) taken from one field is in the same range resulting in relatively large differences (RSD ranges from 20 to 33%) in residues in composite samples of size 5-12. The between-fields and within field variability of residues shall be taken into account in planning monitoring programs and interpreting the results, respectively.

Due to the very large number of agricultural products not all of them can be tested with equal frequency. The monitoring/control programs may be designed taking into consideration, among others, the dietary importance of food item, toxicity and concentration of residues occurring at harvest in supervised trials, prior information from monitoring programs on frequency and level of detectable residues in the marketed commodity, the requirements of plant protection, amount of pesticide sold, etc.

The lecture will present examples for risk based ranking of commodities, and statistical methods for planning sampling programs for detecting specified violation rate and interpreting measurement results.

O-15 The Korean National Residue Program for Veterinary Drugs, Pesticides, and Environmental Contaminants in Food of Animal Origin

MeeKyung Kim, Dong-Gyu Kim, Jin-Young Shin, Kyung Yuk Ko, Chae-mi Lim, Seong-Wan Son

Animal, Plant and Fisheries Quarantine and Inspection Agency, 175 Anyangro, Anyang, Gyeonggido 430-757, Republic of Korea; mkim@korea.kr

Chemical residue analysis is an important role of the national quarantine service to ensure food safety for domestic and imported products. The chemical residue control system in Korea consists of a national residue program (NRP) for domestic products and an import residue testing program for imported products. Residues from veterinary drugs, pesticides, and certain environmental contaminants in food of animal origin are analyzed at the Animal, Plant and Fisheries Quarantine and Inspection Agency and the provincial veterinary services according to an annual sampling plan. The domestic sampling is to prevent illegal on-farm chemical application practices. The import sampling is to verify the quality of exporting countries' residue control programs. The NRP are divided into a monitoring plan, surveillance & enforcement testing, and exploratory projects. The national data from the programs provide the structures to identify and evaluate compounds of health concern. Regulatory actions are taken when violations are found, and results are reported, which are taken into account to make the next activities and plans. The Korean maximum residue limits are established for 110 veterinary drugs, 83 pesticides, and 6 environmental contaminants. Korean delegates are participating in Codex Alimentarius for the international harmonization of regulatory limits and methods of analysis and sampling. International or local inter-laboratory proficiency tests are regularly conducted for analytical quality assurance purposes. In an example event, the Korean monitoring plan contributed globally to investigate the dioxin contamination from the Chilean pork in 2008, and zinc oxide contamination in feed additives was found to be a source.

O-16 Analytical Opportunities in Drinking Water Safety

Wei Wang, Jessica Boyd, Haiying Du, Xing-Fang Li*

Division of Analytical and Environmental Toxicology, Department of Laboratory Medicine and Pathology, Faculty of Medicine and Dentistry, University of Alberta, Edmonton, Alberta T6G 2G3, Canada

Disinfection of drinking water is the most effective public health measure of disinfecting pathogens and of eradicating waterborne diseases. An unintended consequence is the formation of disinfection byproducts (DBPs) from the reactions between disinfectants (e.g., chlorine and chloramines) and natural organic matter in water. To control drinking water quality, surrogate DBPs such as several trihalomethanes (THMs) and haloacetic acids (HAAs) are currently regulated in many countries. Epidemiological studies have shown an association of drinking chlorinated water and increased risk of developing bladder cancer. However, accumulating evidence suggests that the regulated DBPs cannot account for the increased bladder cancer risk observed in epidemiological studies. Majority of DBPs are unidentified; it is proposed that other unidentified yet more toxic DBPs may contribute to the risk.

Our research aims at developing ultrasensitive techniques to identify unknown DBPs that are likely carcinogens. One of such examples is high performance liquid chromatography with tandem mass spectrometry (LC/MS/MS) techniques for characterization of new nitrosamine DBPs, a class of DBPs that are probable human carcinogens. We have demonstrated that the LC/MS method can detect nitrosamines that are not detected by GC/MS. In addition, we identified new N-containing DBPs, and non-chlorinated and chlorinated aromatic DBPs using LC/MS techniques. Recently, we have discovered haloquinones and related compounds as a new class of DBPs that are widely present at ng/L levels in treated water. We will present the recent studies of the occurrence of haloquinones in drinking water systems and *in vitro* toxicity of these DBPs. The high frequency of occurrence and toxicity highlight the importance of monitoring and control of HBQs in drinking water.

O-17 Southern US state laboratories report their chemical residue activities: doing more with less

Sherry T Garris[1], Chanin Craft[1], Jo Marie Cook[2], Loretta Fourrier[3], Nina Huffstetler[3], Gale Hagood[4], Joe Basile[5], and Susan Eigen[6]

[1]**South Carolina Department of Agriculture,** 123 Ballard Court, West Columbia, SC, 29172, USA; sgarris@scda.sc.gov; [2]**Florida Department of Agriculture and Consumer Services,** 3125 Conner Blvd., Building #3, Tallahassee, FL 32399-1650, USA; jomarie.cook@freshfromflorida.com; [3]**Agricultural Chemistry,** Highland Rd, LSU, Baton Rouge, Louisiana 70803, USA; loretta@ldaf.state.la.us , nina@ldaf.state.la.us; [4]**Mississippi State Chemical Laboratory,** 310 President's Circle, Mississippi State, MS 39762, USA; ghagood@mscl.msstate.edu; [5]**Alabama Department of Agriculture & Industries, Food and Drug Laboratory,** 1445 Federal Drive, Montgomery, AL, 36107, USA; joseph.basile@agi.alabama.gov; [6]**Georgia Department of Agriculture,** 19 Martin Luther King, Jr. Dr., Atlanta, GA 30334, USA; Susan.Eigen@agr.georgia.gov

Six US State Laboratories will report on their Chemical Residue Activities, very much like in the early years of the Workshop and from each of their different perspectives. For example, Sherry from SC will talk about doing more analyses with less money and staff. Showing how a very small State Lab can adapt and thrive with method innovation. How even a small lab can still contribute to Food Safety and see emerging chemical residue trends in the food supply (i.e, Pyrethroids).

Jo Marie from FL will talk about how a larger State Lab manages projects and funding from federal and private sources; how they recently renovated, purchased new instrumentation, hired and trained new staff to screen seafood samples for PAH's / dispersants and update on joint work with FDA FERN to screen arsenic in juices.

Loretta/Nina from LA will report on their activities with regard to PAH's and other laboratory issues from the perspective of a State and University Lab.

Gale/Joe from MS/AL will report how State and FDA Labs collaborate in the testing for PAH's in Gulf Coast Seafood.

Susan from GA will report on their activities with regard to testing for antibiotics in feed.

O-18 Data Strategies for HRMS Food Analysis

Walter Hammack, Florida Department of Agriculture, 3125 Conner Blvd, Tallahassee, Fl 32399-1650, USA; Walter.hammack@freshfromflorida.com

The increasing demand for food safety laboratories to screen thousands of samples per year for hundreds or thousands of "targeted" compounds combined with the ever present need to find "unknowns" has resulted in more focus on HRMS instrumentation and the associated processing software. While the current generation of hardware continues to improve by leaps and bounds, it appears that software platforms that can both detect and identify large numbers of compounds in a manageable timeframe still eludes may of the users in the field. With the increasing role that HRMS is playing in the food safety arena, a focus on current software capabilities and limitations are explored.

O-19 Comprehensive Two-Dimensional Gas Chromatography (GCxGC) for Mega Multi-Analyte Methods

Jack Cochran[1], Michelle Misselwitz[1], Julie Kowalski[1], and Mark Merrick[2]

[1]**Restek Corporation**, 110 Benner Circle, Bellefonte, PA, 16823, USA; jack.cochran@restek.com
[2]LECO Corporation, 3000 Lakeview Avenue, St Joseph, MI, 49085, USA

GCxGC is a powerful multidimensional approach to sample analysis that yields superior separation power versus what can be achieved with one-dimensional GC. When paired with a fast acquiring, high sensitivity, full mass-range analysis time-of-flight mass spectrometer (TOFMS), it is unmatched in its potential for mega multi- (GC) analyte methods for target, non-target, and unknown compounds. The benefits of GCxGC-TOFMS include: monitoring/quantifying an unlimited number of target analytes because of the high speed capability of TOFMS, full mass spectra for target compound confirmation purposes, sub- to low- pg sensitivity, mass spectra/identification/semi-quantification of non-target and unknown compounds (including emerging compounds of concern), historical records of mass spectra for future data mining, and the minimization of potential matrix interferences through multidimensional separation. Despite these significant advantages over other instrumentation for the last 10 years, GCxGC-TOFMS

is mostly considered a research tool and has not found widespread routine use in trace residue analysis labs. Although there are numerous reasons for its current position, perhaps the chief one is data processing and review complexity. Still, GCxGC does have its place in mega multi-analyte methods, as will be demonstrated in this talk with several examples, including a recent petroleum biomarker study for crude oil from the Deepwater Horizon oil spill and tarballs collected from the Florida gulf coast.

O-20 The Use of High Resolution Liquid Chromatography/High Resolution Mass Spectrometry in Screening Produce for Pesticide Residues

Brian D. Eitzer[1], Walter J. Krol[1], Walter Hammack[2], Michael Filigenzi[3], Karen Wolnik[4], Lora A. Lin[4] and Sara Robison[4]

[1]**The Connecticut Agricultural Experiment Station**, 123 Huntington St. New Haven, CT 06511; brian.eitzer@ct.gov; [2]Florida Department of Agriculture, Tallahassee, FL; [3]The California Animal and Food Safety Laboratory, Davis, CA; [4]The Food and Drug Administration Forensic Chemistry Center, Cincinnati, OH

The use of liquid chromatography/mass spectrometry in the multi-analyte analysis of pesticide residues in produce samples has become a well-established technique. In most cases analyte specificity is achieved through the use of mass spectrometry/mass spectrometry (MS/MS) monitoring specific parent ion to product ion transitions. High resolution mass spectrometry (HRMS) offers an alternative mechanism to achieve that analyte specificity. The specificity is provided by the use of the exact masses of the residues being monitored. We report here on the use of a Thermo Exactive mass spectrometer operating at a mass resolution of 100,000 with positive ion electrospray in pesticide monitoring programs. Results are shown from a Food Emergency Response Network Chemistry Cooperative Agreement Program interlaboratory study. In this study three produce matrixes were spiked with 250 pesticides at three different levels. The samples were extracted with a QuEChERs based extraction and analyzed with LC/HRMS at 100,000 resolution. In this study 208 of those compounds could be seen in all matrixes and replicates at 200 ppb, and many had much lower detection limits. We also show how the HRMS techniques has impacted the Connecticut pesticide monitoring program, allowing us to expand the number of compounds monitored with over 500 now routinely screened for by LC/HRMS. These data are complementary to our traditional HPLC/MS/MS data, providing confirmation (or ruling out) the presence of residues. Data from selected herb samples are compared to residue data acquired on an Exactive using direct analysis in real time ambient pressure desorption ionization rather than traditional HPLC/MS.

O-21 The Issues with Multiresidue Methods for Pesticide Analysis in Food

Kai Zhang[1], Jon Wong[1], Paul Yang[2], Douglas Hayward[1], Alex Krynitsky[1]

[1] US Food and Drug Administration, Center for Food Safety and Applied Nutrition, College Park, MD 20740, USA; kai.zhang@fda.hhs.gov; [2]Ontario Ministry of the Environment, 125 Resources Road, Etobicoke, Ontario, M9P3V6, Canada

The newly passed FDA Food Safety Modernization Act (FSMA) aims to shift the focus from response to prevention. To prevent contaminated food from reaching consumers, it is vital to improve the capacity to screen various contaminants in foods. As one of the major contaminants in foods, pesticides are routinely monitored to ensure that their concentrations are lower than the Maximum Residue Levels established by EPA, EU and other government agencies. The advent of modern GC-MS and LC-MS technologies has greatly improved the screening capacity of pesticide analysis in foods. Various targeted and non-targeted GC-MS and LC-MS methods have been developed to identify and quantitate a large number of pesticides in a single sample injection. There are challenging issues related to these multiresidue methods such as availability, stability and traceability of the standards, method validation, data analysis, automated sample preparation, matrix effect, identification criteria and harmonization of existing analytical procedures. The presentation will discuss these issues that we have encountered in the course of method development and validation and propose possible alteratives for the future studies. It is worth noting that when including more pesticides, one has to balance the advantages of the increased coverage against the increased complexity which may compromise the usefulness of these multiresidue methods.

O-22 Analysis of pharmaceuticals, steroids and antibiotics using LC-Orbitrap mass spectrometry with enhanced sensitivity, selectivity and minimal matrix effects

Paul Yang and Serei Thach, **Ontario Ministry of the Environment,** 125 Resources Road, Etobicoke, Ontario, M9P3V6, Canada; paul.yang@ontario.ca

Pharmaceuticals and personal care products (PPCPs) are anthropogenic pollutants originating from agricultural and human activities. Similar to modern pesticides, PPCPs are a diverse group of biologically active chemicals that may present a risk to the environment. PPCPs are typically analyzed by liquid chromatography-tandem mass spectrometry (LC-MS/MS) which , to dated has provided the highest selectivity and sensitivity. However, LC-MS/MS can be extremely sensitive to matrix effects as represented by signal suppression/enhancement in the electrospray ionization (ESI) source, resulting potentially biased quantitative analysis. Using quality assurance data and field sample results obtained from routine LC-MS/MS and LC-Orbitrap analysis, selectivity, sensitivity, and matrix effects in PPCP analysis were compared. As expected, severe matrix interferences were observed in the LC-MS/MS analysis of waste water and agricultural run-off samples, revealing problems with specific multiple reaction monitoring (MRM) channels for some target analytes and therefore can result in false-positive identification even with the use of a second MRM transition. The orbitrap has superb mass axis stability and the signal-to-noise ratio (SNR) of target analytes increase with increasing MS resolution. This work

demonstrates that the orbitrap has similar/better instrument detection limits than those obtained from LC-MS/MS analysis and, thus, improved method detection limits. Available full scan HRMS data and accurate mass also afforded the possibility of known-unknown and unknown-unknown identification of PPCP metabolites that may occur during the ozone and/or ultraviolet based treatment technology, providing additional data for toxicity and efficacy studies that cannot be done with LC-MS/MS analysis.

O-23 LC/Q-TOF-MS for the Analysis of Emerging Contaminants in Water Samples

Imma Ferrer and E. Michael Thurman; **Center for Environmental Mass Spectrometry**, Department of Environmental Engineering, University of Colorado, Boulder, CO, USA; imma.ferrer@colorado.edu

Liquid chromatography coupled to mass spectrometry using time-of-flight (LC/Q-TOF-MS) has proven to be an excellent technique for the analysis of emerging contaminants in the environment. The high degree of confirmation by accurate mass measurements for target pharmaceuticals has demonstrated the applicability of the method in routine analysis of several types of water samples. Moreover, the use of high resolution TOF-MS allows for non-target identification, because the full-spectrum is recorded at all times, which is not possible with LC-MS-MS and other MRM techniques. Several strategies, using accurate mass tools by LC/Q-TOF-MS, for the analysis of pharmaceuticals in the environment and their degradation products are reported here. This technique was successful for finding non-targets and unknowns that have not been previously included in routine target methods (lamotrigine, dextromethorphan, gabapentin and some of their metabolites). This talk will present an overview of the main achievements using LC/Q-TOF-MS for identification of new emerging contaminants in water samples.

O-24 Rapid PCB Micro-Extraction of Soil Samples: Method Development

David O Wilson, Lorraine Iverson, Margie St. Germain

U. S. Environmental Protection Agency, 300 Minnesota Avenue, Kansas City, KS 66101, USA; stgermain.margie@epa.gov

Frequently, EPA needs to make quick decisions regarding PCB contamination for emergency responses such as the flooding in Oklahoma where several transformers were compromised, or reduce costs on RCRA site evaluations by using rapid screening samples. Currently, the traditional PCB method would take up to three days to provide very accurate results. What was needed was a rapid method with some accuracy so decisions on protective garments could be made within 24 hours or selections of a subset of samples for full analysis while field crews were available. EPA Region 7 in collaboration with Kansas University developed a rapid, micro-extraction for PCBs in Soil using ordinary laboratory equipment. The initial method was further refined while remaining portable. The method was tested with four different Aroclor mixtures in six different soil samples to check for durability and feasibility. The results show that the method has accuracy of 60-80% recovery and precision of less than 30% Relative Standard Deviation (RSD). The method detection limits are slightly above the traditional method, but below the RCRA action limit of 1 mg/kg. This presentation will show the evaluations of several variations on the method, optimizing the speed as well as accuracy and precision, and durability using several real samples.

O-25 Dioxins and Other Environmental Contaminants in Catfish from U.S. Commerce

Sara J. Lupton,[1] Janice K. Huwe,[1] Emilio Esteban,[2] and Otis Miller;[3]

[1]**USDA, ARS, Biosciences Research Laboratory,** 1605 Albrecht Blvd; Fargo, ND 58102, USA; sara.lupton@ars.usda.gov;
[2]USDA, FSIS, Office of Public Health Science, Athens, GA, USA; [3]USDA, FSIS, OFO, Catfish Inspection Staff, SW Washington, DC, USA

The Food, Conservation, and Energy Act of 2008 (Farm Bill), amended the Federal Meat Inspection Act to provide that "catfish, as defined by the Secretary," is an amenable species and therefore subject to continuous inspection by the USDA Food Safety Inspection Service. Since few background data are available on levels of environmental contaminants in commercial catfish, an assessment of catfish products for residues of dioxins, polychlorinated biphenyls (PCBs), and polybrominated diphenyl ethers (PBDEs) was conducted. Data from this survey will establish typical background levels of these contaminants in domestically marketed catfish and may be used in risk assessments for estimating human exposures. Catfish (N=744) were collected for the 2009-2010 USDA Pesticide Data Program (PDP), and 202 were randomly chosen for dioxin, PCB, and PBDE analysis. Dioxin toxic equivalency (TEQ) concentrations ranged from non-detectable to 3.46 pg/g ww, while PCB TEQs averaged about 10% of the dioxin value. The persistent indicator PCB concentrations ranged from 9.6 – 1383 pg/g ww and were three-fold higher than PBDE sums. All values were lower than those previously reported in a small survey of wild-caught and farm-raised catfish from Mississippi in 2006; however this could be due to geographical or temporal differences or to the smaller sample size of the 2006 survey. The dioxin congener patterns observed in the PDP catfish were similar to those observed in the 2006 farmed catfish as well as catfish exposed to ball clay in the mid-1990s, although TEQs averaged 100-times lower than the ball clay contaminated catfish.

O-26 Using Stir-Bar Sorptive Extraction (Twister™) for PAHs, Pesticides and PCPPs Analysis of Surface Water

Lorraine Iverson, Margie St Germain; **U.S.EPA, Region 7 Science and Technology Center**, 300 Minnesota Ave, Kansas City, KS 64151, USA; iverson.lorraine@epa.gov; stgermain.margie@epa.gov

Traditional sample preparation of water samples for organic analysis in environmental laboratories (such as liquid/liquid) often comes with several drawbacks. It is labor intensive, uses large amounts of toxic solvent, is time-consuming, and needs a relatively large aliquot of the original sample. Region 7 EPA has adopted a newer technique which overcame all of these problems while gaining sensitivity and lower detection limits. Stirbar Sorptive Extraction (SBSE) using Twister™ employs an adsorptive coating on a magnetic stir bar. The technique provides enhanced sensitivity over the traditional extraction techniques because the entire extracted fraction is quantitatively introduced into the GC/MS system by thermal desorption. While stirring the sample, the Twister™ efficiently extracts organic compounds from aqueous matrices. After extracting the samples, each Twister™ is placed into a sealed desorption liner, placed in a thermal desorption unit (TDU), and introduced directly into the GC/MS. Twisters™ work well for neutral compounds that have limited solubility in water. Region 7 used the technique in the summer of 2011 for a continuing ambient monitoring project studying the health of Kansas City urban streams. Replacing traditional extractions and analyses of PAHs and Organochlorine/Triazine Pesticides with Twister™, we saw increases in sensitivity up to 100 fold versus the traditional method. For the first time in six years of data, certain PAHs and pesticides were detected in urban Kansas City streams. Additionally, we added certain personal care products and pharmaceuticals to the target list. Successes and struggles involved with the implementation of the new technique will be discussed.

O-27 Reference Material Database provided by the TDRM of AOAC INTERNATIONAL

John Budin,[1] and Donna Zink;[2]

[1]**Mérieux NutriSciences Corporation**, 111 E. Wacker Dr. Ste 2300, Chicago, IL 60601 USA; john.budin@silliker.com;
[2]AIM Research Enterprises LLC

The Technical Division on Reference Materials (TDRM) of AOAC INTERNATIONAL recommends policy and criteria to facilitate development and use of reference materials in validation, implementation and routine use of AOAC methods. As part of these goals, TDRM has developed an online database for maintaining searchable information on reference materials by analyte or matrix, with a third option for searching reference materials that may be suitable for use with an OMA (AOAC Official Methods of Analysis) method based on analyte and method scope. Only essential information is included to maximize usefulness and minimize effort required to keep information current. Specific information such as measurement uncertainty will be available through a link to the producer's certificate, when that information is available online. Data that is submitted by national metrology institutes and accredited producers is included. The data is repopulated annually at the end of each year. The project was developed using an Access database to build a model, with input from TDRM Members, for up to date, broadly-accessible internet database system. The current status of this project is that the online database has been developed with funding support from the NIH Office of Dietary Supplements, and the period for review and input by TDRM members is completed. The year 2012 is the first year that comprehensive sets of data are being request from producers to launch the database for full use. The ultimate objective of the TDRM is to make this database broadly accessible to the general public. The database will be presented with examples.

O-28 The Challenges Faced during Development and Validation of Methodology Based on Liquid Chromatography–Tandem Mass Spectrometry (LC-MS/MS) for the Monitoring for use of Anabolic Steroids in Animals

Simon Hird and George Stubbings

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

A wide range of anabolic steroids has been used in animal fattening because of their capacity to increase weight gain and the improvements in feed conversion efficiency. The use of anabolic steroids in animal fattening is prohibited in the European Community and monitoring for use of anabolic steroids is carried out through the National Plans of the individual Member States. For controls at retail level and for products imported in the EU, it is necessary to have analytical methods applicable to meat samples, whereas at farms and abattoirs, misuse of anabolic steroids in living animals is monitored by analyses of the animal's urine and/or serum. Because of the complexity of these matrices and the low "Recommended Concentrations" established, it is necessary to have sensitive, selective and specific methods for the detection of the anabolic steroids.
Methodology has been developed for the determination of 15 anabolic steroids in bovine, ovine and porcine urine. The procedure involved enzymatic hydrolysis prior to extraction and SPE prior to analysis by ultra high performance liquid chromatography coupled to a tandem mass spectrometer operating in electrospray, switching between positive and negative ion modes. Data acquisition was performed in selected reaction monitoring (SRM) mode. The method was validated according to the Commission Decision 2002/657/EC, for the detection and confirmation of residues in products of animal origin. The methodology developed is suitable for the detection, quantification and confirmation of identity of these anabolic steroids in bovine, ovine and porcine urine and can be used for residue control programs.

O-29 Use of APPI to Minimize ESI Matrix Effects in LC-MS Analysis of Pesticides in Foods

Robert Voyksner,[1] Sheng-Suan Cai,[2] Jennifer Voyksner,[1] Brian Nies,[2] and Jack Syage[2]

[1]**LCMS Limited**, 1502 West NC-54, Durham, NC 27707; robert_voyksner@lcmslimited.com
[2] Syagen Technology, Inc., a wholly owned subsidiary of Morpho Detection, Inc. 1411 Warner Ave, Tustin, CA 92780

ESI has been the most popular ionization technique used for LC-MS analysis of pesticides. However, ESI fails to efficiently ionize low proton affinity pesticides containing electron drawing groups and suffers severe matrix effects for analysis of complex sample matrices. In contrast with ESI, APPI is able to simultaneously ionize both high and low proton affinity small molecules with high sensitivity and is much less susceptible to ion suppression. This work compares APPI and ESI for the LC-MS analysis of 16 pesticides that have been a challenge for LC/MS in vegetables and fruit.

To achieve the best sensitivity, dopants were added for APPI. Chlorobenzene was best for positive ion, while toluene was best for negative ion. APPI produced $[M+H]^+$ ions for most pesticides in positive ion mode and M^- or $[M-X]^-$ ions in negative ion mode, (where x = methyl, ethyl or ethoxy groups). ESI produced $[M+H]^+$, $[M+Na]^+$, $[M-H]^-$ and $[M+acetate]^-$ ions.

Matrix effects for APPI and ESI were evaluated using homogenized tomatoes, apples and carrots with gradient elution run times of 3, 10 and 20 minutes. The average percent recoveries for these 3 experiments are as follows:

Gradient (min)	recovery APPI			recovery ESI		
	Range(%)	Mean	std dev(%)	Range(%)	Mean	std dev(%)
3	52-117	84	11	4-522	106	137
10	65-128	90	10	2-505	81	59
20	79-137	105	22	3-485	78	50

These results indicate that reduced matrix effects offered by APPI significantly simplifies sample cleanup procedures and shortens the time required for on-column LC separation.

O-30 Analysis of Emerging Environmental Contaminants: Where We are Now, and Thoughts About Where We Should Go

Yelena Sapozhnikova[1], Stephen Timko[2], Edward Wirth[3], Michael Fulton[3] and Steve Lehotay[1]

[1]**US Department of Agriculture, Agricultural Research Service, Eastern Regional Research Center**, 600 East Mermaid Lane, Wyndmoor, PA, 193038, USA; yelena.sapozhnikova@ars.usda.gov; [2]University of South Carolina, Columbia, SC; [3]Center for Coastal Environmental Health and Biomolecular Research, NOAA, NOS, NCCOS, Charleston, SC.

This presentation will outline the current trends and challenges in method development and analysis of emerging environmental contaminants. Advances in analytical chemistry and instrumentation enhance our analytical capability to identify and measure these contaminants in various matrices. However, their effects on human health and the environment in most cases are lacking. As a part of NOAA's Oceans and Human Health Initiative we analyzed seawater for artificial sweeteners including sucralose (Splenda), and measured up to 585 ng/L of sucralose in surface water from Charleston Harbor, SC. Additionally, farmed and wild shrimp from the USA and other countries were analyzed for synthetic musk fragrances and up to 762 ng/g lipid weight of musk Galaxolide was measured in shrimp. Recent USDA research efforts involved the development of a multi-class, multi-residue method for the analysis of novel flame retardants, representative pesticides, PCB and PBDE congeners and PAHs (67 analytes total) in catfish muscle. Other potential emerging contaminants include chemicals migrating from packaging materials, industrial pollutants, perfluoro-hydrocarbons, silicones, nanoparticles and other not previously recognized contaminants. Most of these chemicals are persistent, can be bio-accumulative and have known or potential impact on human health. What emerging environmental contaminants need to be monitored, and should we conduct targeted analysis or non-targeted screening to identify potential contaminants missing in targeted analysis? The goal of this presentation is to stimulate a discussion among scientists on what emerging contaminants are of concern in food and environmental samples, and how to prioritize method development and monitoring needs.

POSTER ABSTRACTS

P1 **A Compound-based Approach to Simplify GC-MS/MS Method Development for 280 Pesticides in Food Matrices**

Ed George,[1] Keyu Zhou, Helen Sun, and Kefei Wang

[1]**Bruker Daltonics Inc.- Chemical and Applied Markets**, 3500 West Warren Avenue; Fremont, CA 94538, USA; ed.george@bruker.com

Tandem mass spectrometry coupled to chromatography, such as GC-MS/MS, operated in MRM mode, is rapidly becoming the method of choice for targeted screening of multi-residue analysis in complex food matrix samples. This is due to its high specificity and capability of simultaneously monitoring of hundreds of MRMs from a large number of compounds in complex sample matrices such as food and environmental samples. However, MRM method development, including setting up the MRM acquisition table and data processing, is complicated and time consuming.

In this study, a new compound-based screening (CBS) approach is introduced and demonstrated for the analysis of 280 pesticides in food matrices on a Bruker SCION TQ GC-MS/MS system. The CBS- based algorithm was designed to significantly simplify MRM method development and data processing by automatically linking data acquisition and processing together, eliminating the need for separate method set-up. The MRMs for the target pesticides can be chosen from a factory library containing hundreds of pesticides along with their retention times from a standard GC method. CBS is able to optimize the dwell time for each MRM for each compound without the use of chromatographic segments. Moreover, the retention times can be auto-adjusted to account for the variation of method parameters such as column length, carrier gas flow rate, and oven temperature programming, making it convenient and flexible to be adopted by different users.

P2 **Children's Food Project – Survey of Pesticide Residues in Infant Foods**

Angela Murray[1], Andrea Leclair[1], Leigh Miller[1], Henri Bietlot[1], and Jian Wang[2]

[1]**Canadian Food Inspection Agency**, 1400 Merivale Road, Ottawa, ON K1A 0K9, CANADA; angela.murray@inspection.gc.ca; [2] Canadian Food Inspection Agency Calgary Laboratory, 3650 36 Street NW, Calgary, AB T2L 2L1, CANADA

The Children's Food Project (CFP) is an initiative of the Canadian Food Inspection Agency (CFIA) to undertake monitoring of pesticide residues in foods consumed by and targeted to children. The CFP provides a snapshot of the nature and levels of pesticide residues found in these foods. This paper examines the levels and types of pesticide residues found in infant foods (i.e., 0 – 2 years), frequency of pesticide residues per sample and compliance rate (to Canadian standards) as determined using multi- and single-residue analytical methods. The methods employed allow for one sample to be tested for up to over 400 different pesticides. A total of 382 and 879 domestic and imported infant food samples were analysed from years 2008-2009 and 2010-2011, respectively. Samples included products such as baby cereal, infant formula, pureed fruits and vegetables, baby cookies, etc. In each of the years approximately 25% of the samples contained detectable levels of pesticide residues with one sample in violation of paragraph 4(d) of the *Food and Drugs Act*. The frequency of pesticide residues per sample were similar in each year with ~9% containing two to five pesticide residues per sample in 2008-2009 and ~10% containing two to seven pesticide residues per sample in 2010-2011. Captan, carbendazim, chlorpropham, and diphenylamine were the most prevalent pesticide residues detected common in both years. The data obtained from studies like the CFP are instrumental in the assessment of the dietary exposure to pesticide residues in foods consumed by Canadian children.

P3 **Automated extraction and sample cleanup for GC of Chlorinated Pesticides by EPA Method 8081**

Tom Hall and Jim Westmoreland, SCI-CON/FMS, 631 Executive Drive, Winter Park, FL, 32789, USA; jimwscicon1@cs.com

Pesticides are classes of chemicals that are used to treat or control outbreaks of pests, especially insects. Food production as well as safety and health organizations heavily rely on pesticides to increase food yields and / or control disease outbreak spread by insects. Some pesticides have proven hazardous to other animals or the environment and have either been restricted or banned. The need to monitor products, especially foods, for pesticides is essential as more pesticides are being discovered to have adverse effects. One class of commonly used pesticides is chlorinated pesticides. These compounds are commonly detected using EPA Method 8081. This method determines the concentrations of various organochlorine pesticides in extracts from solid and liquid matrices, including food products, using gas chromatography with electron capture detectors (ECD). This application provides a unique new option to speed up multiple sample prep processes into one. The FMS PLE (pressurized liquid extraction) system will automatically perform the extraction and sample cleanup using the FMS proprietary in cell column and deliver the extract for final concentration to be injected directly by the GC system. This is a first for analyzing chlorinated pesticides by EPA Method 8081.

P4 Analysis of 136 Pesticides in Avocado using a Modified QuEChERS Method with LC-MS/MS and GC-MS/MS

Narong Chamkasem[1], Lisa W. Ollis[1], Tiffany Harmon[1], Greg Mercer[2]

[1]**US FDA, SRL**, 60 8th St. NE, Atlanta, GA, *narong.chamkasem@fda.hhs.gov*
[2]US FDA, PRLNW, 22201 23rd Dr. SE, Bothell, WA

A simple, high-throughput method for the analysis of 136 pesticides in avocado by LC-MS/MS and GC-MS/MS is presented. A modified QuEChERS sample preparation method employing high solvent to sample ratio was developed to improve the extraction recovery of very lipophilic pesticides. After acetonitrile extraction and salting out step, one µL of sample extract was injected directly to LC-MS/MS. The recovery and relative standard deviation for all analytes quantified by LC-MS/MS method at 10, 50, and 200 ng/g spiking levels are excellent at 107.12 ± 9.25, 86.05 ± 6.73, and 94.77 ± 4.23, respectively. For GC-amenable only pesticides, one mL of acetonitrile extract is pipetted into a 2-mL centrifuge tube containing 150 mg anhydrous $MgSO_4$, 150 mg primary secondary amine (PSA) sorbent and 50 mg C-18 sorbent. The tube is mixed and centrifuged to absorb fat residue in the extract. The extract is injected into a gas chromatograph/tandem mass spectrometer (GC-MS/MS) using temperature programmable injector and column back flush program. The excellent sensitivity of GC-MS/MS allows injection of diluted sample to detect the pesticides in fatty sample without sample concentration. The recovery and RSD for 57 analytes quantified by GC-MS/MS method at 10, 50, and 200 ng/g (n=5) are adequate at 73.94 ± 20.30, 70.15 ± 11.35, and 94.35 ± 18.05, respectively. The method has improved the recovery of the problematic lipophilic pesticides in the QuEChERS method and has been useful for the screening efficiency and throughput of pesticides.

P5 Rapid Screening for Multiclass Pesticides in Meat at the USDA -Food Safety and Inspection Service (FSIS).

Son Hoang, Sabrina Leung, Nhung Nguyen, Megan Kennedy, Sushma Srivastava, Catalina Yee.

USDA - FSIS - Office of Public Health Science - Western Laboratory: 620 Central Ave. Bldg. 1, Alameda, CA 94501, USA; Son.Hoang@fsis.usda.gov

Exposure to pesticides can cause health problems, such as neurological effects, birth defects and cancer. One route of exposure is through food, in which ingested pesticides accumulate in the fat deposits in the body where they remain and cause damage. In May 2011, FSIS refocused its laboratory testing on pesticides of highest public health concern. The program expanded the number of pesticides monitored from 44 to 56, and increased the number of pesticide classes covered from 1 to 9. The analytical screening method detects pesticides at part per billion (ppb) concentration levels in meat. The extraction method was developed by the USDA Agricultural Marketing Service using QuEChERS approach with ultra-low temperature and C18 Solid Phase Extraction to minimize co-extracted lipids. Samples were analyzed by LC/MS/MS and GC/MS/MS. Concentration estimates were calculated using a one-point matrix matched calibration. Fortification levels were at 5–80 µg/kg and recoveries fell within 60-125% for 95% of the 56 analytes. The screening of 2,247 samples, followed by confirmation and quantitation of screened positives, led to the identification of two violations: piperonyl butoxide (117 ppb) and carbaryl (49 ppb). Five additional samples contained piperonyl butoxide, but the concentrations were belowed tolerance levels. This illustrates the capability of FSIS to protect the public health by extending the method to a total of 96 analytes from 13 classes. The analytical method provides a more complete picture of the pesticides in the commercial meat supply and eliminates a number of the UARs that were detected previously but undefined.

P6 Strategies for Building Broad Multi-Residue Screens by LC/GC-Mass Spectrometry

Kelly Dorweiler,[1] Laura Marshak,[2] Vishwas Ghatge;[2]

[1]**General Mills/Medallion Laboratories** 9000 Plymouth Ave Golden Valley, MN 55427, USA; Kelly.Dorweiler@genmills.com
[2] General Mills/Medallion Laboratories 601 Prudential Hiranandani Gardens Powai, Mumbai, Maharashtra 400053, India

The need for more comprehensive pesticide screens is growing. Strategies to efficiently expand existing screens while balancing sample queues requires a systematic and streamlined process. The strategy we have followed to expand and implement our screens, and subsequently test hundreds of pesticides in foods within an accredited ISO 17025 laboratory will be discussed.

The QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) technique has been employed to extract a wide range of chemical groups, coupled with liquid and gas chromatography-mass spectrometry for analysis. Early evaluation was performed using mushroom and flaxseed data. Of the 540 pesticides evaluated by two variations of AOAC 2007.01, 405 pesticides met our laboratory's acceptance criteria. In mushrooms, 86% of the analytes were recovered 80-120%. A decrease in recovery was observed in flaxseed, which was expected due to higher lipid content.

Further preliminary validation was performed through fortification experiments of dry grains, ready to eat cereals, and legumes. Pesticides were extracted most favorably in rice, with 348 analytes recovered between 70% and 120%. Legumes recovered 34 fewer pesticides. This was attributed in part to poorer extractability of legumes and later eluting chromatographic interference. Pesticides with lower recoveries remained in scope for all matrices, provided analyte response was sensitive and selective to ensure successful identification.

P7 Standard Response Stability of Trace Pesticides in QuEChERS Cantaloupe Extracts: An Inlet Comparison

Ryan C. Venable and Raymond A. Allum

Bureau of Chemical Residue Laboratories, Division of Food Safety, Florida Department of Agriculture and Consumer Services, Tallahassee, Florida 32399, USA; ryan.venable@freshfromflorida.com

Pesticide residue laboratories are challenged to analyze as many samples as possible for as many different pesticides as possible in as little time as possible. Innovations in gas chromatography and mass spectrometry have allowed food safety laboratories to measure hundreds of compounds at part per billion concentrations in a single run. This study illustrates the effects of QuEChERS cantaloupe extract on instrument response stability to external standards in matrix during sets of 25 cantaloupe samples. Each set of extracts and external standards was analyzed before and after a 1:2 dilution with acetonitrile and with acetone. Each set was also analyzed using two popular GC inlets, a Gerstel Programmed Temperature Vaporization inlet and an Agilent Multi-Mode inlet in Solvent Vent mode installed on two Agilent 7890 GCs coupled to Agilent 7000 Triple Quadrupole Mass Spectrometers. Changes in sensitivity to 94 pesticides were measured throughout each of the sample sequences. No significant differences in response stability trends were observed of the combinations of inlet types and sample matrix alterations.

P8 Monitoring of pesticides residues in Paprika Using GC-MS/MS from Gyeongnam region in Korea

Mi yeong Kim, Mun Hee Yun, Seung Hwa Lee, Yu Jeong Nam, Min Ho Park, Hyun Min Jang, so yeong Park;

[1] **Gyeongnam Provincial Office, National Agricultural Products Quality Management Service (NAQS**), 214 Geoje-daero, Yeonje-gu, Busan, 611-804, South Korea; aquarubby@korea.kr

Monitoring pesticide residues in agricultural commodities is the key to meet the international regulations and to enhance international competitiveness of Korean agricultural commodities. 80 kinds of pesticides were monitored in 139 paprika samples collected from Gyeongnam area. The pesticide residues were extracted by EN15662 buffer based on the QuEChERS method, clean-upped with dispersive solid-phase extraction method, and analyzed using GC-MS/MS. From these results, tetraconazole, chlorfenapyr, acrinathrin and azoxystrobin were most frequently detected, and 12 pesticides were detected in 70.5% of the paprika samples. Although detection frequencies were high, all samples complied with the maximum residue limits (MRLs) set by both the Korea Food and Drug Administration (KFDA) and Japanese Ministry of Health, Labour and Welfare.

P9 Determination of Maleic Hydrazide Residue in Tobacco by LCMSMS and the QuEChERS

Meng Yu, Tim J. Trees, and Chuck J. Neslund

Lancaster Laboratories, Inc. , 2425 New Holland Pike, Lancaster, PA 17605, USA; myu@lancasterlabs.com

Maleic hydrazide is a plant growth regulator that is applied to crops to reduce sprouting and the growth of suckers in tobacco, potatoes, and storage bulb vegetables such as onions. In the U.S., about 85% of maleic hydrazide is used in the tobacco industry. Analysis of maleic hydrazide residue in tobacco is challenging due to its low molecular weight (112), matrix complexity from drying, and glucoside complexation which requires an acidic or basic hydrolysis treatment step. A highly pigmented and complicated extract is therefore generated. The QuEChERS sample preparation and cleanup technique is widely used for many pesticide compounds and matrices, but application to maleic hydrazide has never been reported. A new LCMSMS method based on ESI negative ion mode was developed after QuEChERS cleanup by PSA/C18/GCB. The LCMSMS chromatogram shows no interference from matrix background, which is not possible with LC UV methods because of the inherent lack of specificity. Two commercially available QuEChERS containing different amounts of GCB were tested for cleanup efficiency and recovery. Critical factors that influence recovery and robustness were studied and optimized. The LCMSMS method was validated in terms of MDL, recovery, and reproducibility. Six point calibration curves of matrix matched standards routinely show good linearity with R^2 >0.995. Triplicate spike recoveries at three concentrations for the two QuEChERS ranged from 73.7% - 100% with %CV of 11, 3.6, 3.3(n=18), respectively. The LCMSMS run requires only 15 minutes. This new method is highly efficient for its simplicity and specificity.

P10 Results for a Pesticide Monitoring Program of German Malts

Mehmet Coelhan and Fritz Jacob

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

The German Brewing industry has more than 1300 members and annualy produces around 100 million hectoliter beer. Although, in Germany, the annual consumption of beer per capita has stagnated for many years at about 110 liters, it is still relatively high in comparison to other European countries. A large part of the beer produced in Germany is exported. German brewers take a great deal of care to ensure that the beer they produce is entirely safe. Pesticides and other plant protection products used in Germany are subject to stringent legislation under German and EU law. The monitoring of beer and malt is also organized by the Association of

German Brewers to check for any contaminants or pesticides. In 2011, a malt monitoring program was started in Germany to explore levels of heavy metals, mycotoxins, dioxins and dioxine-like PCBs, and a large number of pesticides (>330). In the present study, the results for pesticide monitoring of German malts (N=30) are presented. Analyses revealed that, among 340 pesticides sought, only very few of them are present in low levels (<0.1 mg/kg).

P11 Effects of Including or Removing of Sweet Corn Silk on the Levels of Pesticide Residues in Flesh Sweet Corn Samples

Tomonari Yajima, Kazuaki Iijima, Masahiro Fujita, Kiyoshi Sato, and Yasuhiro Kato

The Institute of Environmental Toxicology (IET), 4321, Uchimoriya-machi, Joso-shi, Ibaraki 303-0043, Japan; iijima@iet.or.jp

The purpose of this study is to provide the relation of residue levels determined by the different portions to be analysis for sweet corn. Because some of the guidelines have a different or obscure definition for the portions to be analysis, such as how to treat the style (silk) of sweet corn. The effects of including or removing of sweet corn silk (style) on the levels of pesticide residues were investigated for 5 pesticides in preharvest flesh sweet corn samples. Sweet corn samples were divided into cob, kernel, silk and husk, which were separately acquired and homogenized for the pesticide residue analysis. The majority of pesticide residues existed in the portions of the husk (94-100%) from sweet corn harvested in the Chiba field. Meanwhile, the pesticide residues existed distributed in the two portions of the silk (45-100%) and husk (<1-46%) in the Ibaraki sample. These results suggest that how to treat the silk of sweet corn for the sample preparation process has sometime significant influenced for the determination of pesticide residue levels.

P12 The QuEChERS Sample Preparation Approach with Dispersive and Cartridge SPE Cleanup, GCxGC-TOFMS, and LC-MS/MS for the Analysis of Pesticides in Tobacco

Michelle Misselwitz, Jack Cochran, Julie Kowalski, and Sharon Lupo

Restek Corporation, 110 Benner Circle, Bellefonte, PA, 16823, USA; michelle.misselwitz@restek.com

The production of tobacco, a high-value crop for the United States, is increased by the use of pesticides that are specifically approved for use on tobacco by the Environmental Protection Agency (EPA). Even after the processing of tobacco, some pesticide residues remain on the product and under its pesticide registration program EPA is charged with assessing risks to smokers from exposure to these residues. Because tobacco is such a complex matrix, the challenges associated with determining pesticides at trace levels are substantial. This includes pesticide extraction from tobacco, and the instrumental analysis step, which can be complicated by matrix interferences.

We used the Quick – Easy – Cheap – Effective – Rugged – Safe (QuEChERS) sample preparation approach with to isolate pesticide residues from tobacco leaf, ranging from such small, polar pesticides such as methamidophos, to relatively nonvolatile, non-polar, large pesticides like deltamethrin. We also explored two extract cleanup methods, dispersive solid phase extraction (dSPE) and cartridge SPE (cSPE), monitoring their efficiency at pesticide recovery and matrix reduction. Comprehensive two-dimensional gas chromatography with time-of-flight mass spectrometry (GCxGC-TOFMS) and liquid chromatography with tandem mass spectrometry (LC-MS/MS) were compared and contrasted for their ability to determine pesticide residues in the resulting extracts.

P13 Multi-Residue Pesticides Analysis in Herbal Tea Products by GC-MS/MS

Hans-Joachim Huebschmann,[1] Joachim Gummersbach,[1] David Steiniger,[1] Nicole Rueckert,[2] Johann Kirchner,[2] Elmar Haefner[2]

[1]**Thermo Fisher Scientific**, 2215 Grand Avenue Parkway; Austin, TX 78728, USA; david.steiniger@thermofisher.com
[2]Phytolab GmbH & Co KG, Vestenbergsgreuth, Germany

The residue analysis of pesticides has developed in recent years into a comprehensive methodology for the detection of many hundreds of potential contaminating compounds. A multiresidue method for teas and herbal products is faced with additional challenges from the worldwide origin of the products and the complex matrix of the dried materials. The dried leaves, fruits or seeds and other medical products deliver highly complex extracts due to the rich content on active ingredients, essential oils and the typical high boiling natural polymer compounds from broken cells, leaves or fruit skins. This application describes the methodology used for the multiresidue pesticides analysis of herbal products using a short sample preparation for quantitation by GC-MS/MS. The data acquisition is using the unique timed-SRM allowing for the detection of a virtually unlimited number of pesticide compounds in one run without sacrificing the high sensitivity for individual compounds. The routine screening method is applied for a wide variety of different sample type ranging from regular black tea or sage leaves, to seeds like fennel and herbs of medical and fragrance use like thyme and chamomile. The data processing and reporting is achieved by using the dedicated TraceFinder quantitation software. The method presented is well prepared for routine analysis, provides high robustness of the chromatographic system and MS ion source reducing the need for frequent maintenance. The method is easy to use in routine, durable and robust even with the most challenging sample types, and fully automated in automatic sampling to found and not-found report generation.

P14 Quantification of pesticides residues in tomato samples using a fast multi-residue method by liquid chromatography tandem mass spectrometry combined with QuEChERS method

Graziela C.R. Moura-Andrade, Sérgio H. Monteiro, Alyne S. da Silva, Jeane G. Francisco, Leila A. Figueiredo, Rafael G. Botelho, Franz Z. Vilca, and Valdemar L. Tornisielo;

Universidade de São Paulo, Centro de Energia Nuclear na Agricultura, Av. Centenário, 303; Piracicaba, SP 13416-903, Brasil; grazielacrm@cena.usp.br

A method for simultaneous identification and quantification of 46 pesticide residues in tomato with column with small particles (1.8 μ m) in 13 min was developed and validated. Samples were extracted and cleaned up with QuEChERS method and detected by liquid chromatography tandem mass spectrometry (LC–MS/MS) under dynamic multiple reactions monitoring mode, and quantified by matrix-match calibration. The validation study was carried out following DG SANCO/2011/12495 of the European Quality Control Guidelines. The linearity of the calibration was between 10 and 1000 ng ml^{-1} concentration ranges, and the limits of quantification (LOQs) less than 0.01mg kg^{-1} for most pesticides. The mean recoveries of pesticides were in the range from 87% to 116% at three concentration levels ranging from 0.01; 0.1 and 0.5 mg kg^{-1} with relative standard deviations (RSD) below than 17%. The method was applied on 51 real tomato samples collected from Piracicaba, SP - BRASIL markets in the first trimester of 2012 and all the residue levels of pesticides were below the MRL required by BRASILian government. This methodology combines the advantages of QuEChERS, columns with small particles and LC-MS/MS producing rapid, sensitive, and reliable results which can be applied in routine analytical laboratories.

P15 Market Basket Survey of Organochlorine Pesticide Residues in Herbs and Spices Using EPA 1699

Martha Maier, Andrew Patterson, Doug Selby, And Mel Schuld

Vista Analytical Laboratory, 1104 Windfield Way; El Dorado Hills, CA 95762, USA; anp@vista-analytical.com;

Organochlorine (OC) pesticides are now entering the seventh decade of global use; current OC pesticides include Endosulfan and Lindane (gamma-HCH) among others. While bans on many OC pesticides have been in place for over thirty years, the persistence of these compounds in the environment is an ongoing issue. EPA Method 1699: Pesticides in Water, Soil, Sediments, Biosolids, and Tissue by High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS) was released in 2007 and covers many current and legacy pesticides. This method also proves amenable for the extraction of herbs and spices. As herbs are grown in many regions of the world, pesticide use is difficult to track especially when dealing with multiple countries of origin. This survey of dried herbs and spices attempts to find patterns in the occurrence of pesticides within the same crop type and between products that were organically farmed verses conventionally farmed. Although vegetation can be a complex matrices, detection at low part-per-trillion levels was possible using multiple column chromatography techniques. Additional isotopically-labeled standards were added above the method requirements to aid in loss-corrected quantitation. Current testing is still underway, but positives of 26 out of 30 OC pesticides have been confirmed with concentrations upwards of 0.5ppm.

P16 Rapid Method for the Confirmatory Analysis of Paraquat and Diquat in Potatoes and Cereals

Diana I. S. Kolberg,[1] Dorothea Mack,[1] Michelangelo Anastassiades,[1] Michael T. Hetmanski,[2] Richard J. Fussell,[2] Thijs Meijer,[3] Hans G.J. Mol;[3]

[1] 1Chemisches und Veterinäruntersuchungsamt Stuttgart, Schaflandstr. 3/2, 70736 Fellbach, Germany; Diana.Kolberg@cvuas.bwl.de
[2]Food and Environment Research Agency, York, UK; [3]RIKILT- Institute of Food Safety, Wageningen, Netherlands;

Paraquat and diquat are broad spectrum contact herbicides used in more than 100 different non-EU countries. In the European Union (EU) diquat is used as a crop desiccant, but the use of paraquat is banned. The EU Maximum Residue Limits (MRLs) for paraquat are set at 0.02 or 0.05 mg kg^{-1}, de-pending on the commodity. The MRLs for diquat are in most cases, set at the "LOQ" (0.05 or 0.1 mg kg^{-1}), but higher MRLs are established for oilseeds (e.g. 5 mg kg^{-1} for linseed); cereals (e.g. 10 mg kg^{-1} for barley) and lentils (0.2 mg kg^{-1}). The traditional analysis of paraquat and diquat involves extraction based on reflux with sulfuric acid, SPE clean up, derivatisation (if required) and quantification by HPLC or GC. In this work a simplified method has been developed based on extraction with a mixture of methanol/water/hydrochloric acid at 80˚C and analysis by LC-MS/MS using isotope dilution. Details of the method are provided in the EURL QuPPe method (http://www.crl-pesticides.eu/library/docs/srm/meth_QuPPe.pdf). Results from both, a small -scale inter-laboratory trial and from the surveillance of potatoes, cereals, pulses and cereal-based baby foods will be presented. The frequency of residues of diquat emphasises the need for laboratories to monitor specific crops.

This work was done as part of the EU project CONtaminants in Food and Feed: Inexpensive DEtec-tioN for Control of Exposure (CONffIDENCE,) which is funded under the European 7th Framework program (FP7-211326-CP). http://www.conffidence.eu/index.php

P17 Detection of Underivatized Glyphosate and Similar Polar Pesticides in Food of Plant Origin by LC-MS/MS

Rolf Kern[1], Stephen Lock[2], Hermann Unterluggauer[3]

[1]**AB SCIEX**, 353 Hatch Dr, Foster City, CA, USA; Rolf.Kern@absciex.com; 2AB SCIEX, Warrington, Cheshire, UK; [3]Austrian Agency for Health and Food Safety (AGES GmbH), Innsbruck, Austria

Glyphosate is a common broad-spectrum systemic herbicide used widely to kill weeds especially annual broadleaf weeds and grasses known to compete with crops. Usually Glyphosate, as it is very polar, undergoes FMOC derivatization by reacting the native Glyphosate with Fluorenylmethyloxycarbonyl chloride (FMOC-Cl) before analysis. This derivatization step complicates the analysis and there is a growing need for a method which can detect not only Glyphosate but also it's major metabolites, for example Glufosinate, in their underivitized state. In addition a simplified approach to sample extraction using either QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) or a solvent extraction would be beneficial.

Here we present initial data using a new LC-MS/MS method which combines the use of a HILIC type chromatography with *Scheduled* MRM™ on an AB SCIEX QTRAP® 4500 LC/MS/MS system to detect underivatized glyphosate and its metabolites which have been spiked in different food matrices. A simple solvent extraction has been used and initial data will be presented to show how applicable this approach is to food analysis.

P18 Multiresidue Analysis of Rodenticides in a Food Matrix Using an Alkaline Extraction and Ultra Performance Liquid Chromatography/Tandem Mass Spectrometry

Lynda Podhorniak and Yaorong Qian

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

Rodenticides are used to control rodent populations but non-target animals can be poisoned inadvertently by ingesting either the treated feeds or the poisoned rodents. Humans are also poisoned by rodenticides which are among the most frequently used chemicals in deliberate self-poisonings and in childhood poisonings. Historically, rodenticides have been analyzed by high-performance liquid chromatography (HPLC) with ultra-violet (UV) or fluorescence detection. Recently, several studies reported the analysis of rodenticides using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS) in a variety of samples, which improved accuracy and detection limits for some of these compounds. The objective of this study was to accurately analyze eight coumarin and indandione anticoagulant rodenticides (coumafuryl, warfarin, diphacinone, chlorophacinone, bromadiolone, and brodifacoum) and a non-anticoagulant rodenticide (bromethanlin) in an egg sandwich using LC/MS/MS. Different extraction techniques were explored to achieve improved recovery and detection of all eight compounds. Those techniques included a neutral and acidic QuEChERS approach extraction and the alkaline extraction of the Food Emergency Network method. The alkaline extraction produced the best results for all compounds with recoveries between 70 to 130% whereas several compounds were not recovered during neutral acetonitrile extraction. Extraction with acidic acetonitrile generated acceptable recovery only for some compounds.

P19 The Use of Microflow UHPLC as a Way to Reduce Solvent Usage in Pesticide Screening of Food Samples by LC-MS/MS

Stephen Lock[1], Pam Stoddart[1], Brent Lefebvre[2]

[1]AB SCIEX, Warrington, Cheshire, UK; [2]**AB SCIEX**, 71 Four Valley Dr, Concord, ON, Canada; Brent.Lefebvre@absciex.com

Traditionally in pesticide screening of food samples, sample are prepared using generic extraction procedures, like QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) and then analyzed by LC/MS/MS or GC/MS/MS. Usually when analyzing samples, LC flow rates are in excess of 500 µL/min and are used in combination with high pressures with smaller particle size HPLC columns to maintain sharp peaks and fast chromatography. These flow rates produce fast speeds and excellent peak shapes and results, but have a draw back in that they require higher volumes of organic solvent. The consumption of HPLC organic solvents, such as acetonitrile and methanol, is a growing cost of analysis and its disposal has an environmental impact. Therefore, ways to reduce solvent consumption in pesticide residue testing will be beneficial to the environment and reduce running costs of a testing laboratory.

Here we present new data using microflow LC (using an Ekspert™ microLC 200 running at 10 µL/min) in combination with a LC-MS/MS method developed on an AB SCIEX QTRAP® 4500 system utilizing the *Scheduled* MRM™ algorithm in combination with fast polarity switching and acquisition of MS/MS spectra for compound identification. Initially this approach has been applied to a screen of over 100 pesticides in QuEChERS food extracts and we will present robustness and sensitivity data on several matrices to show its applicability in food analysis.

P20 A High-Throughput Approach to Multi-Matrix Food Testing Using QuEChERS and Tandem GC/MS

Joan Stevens,[1] Melissa Churley[2]

[1]**Agilent Technologies**, 25200 Commercentre Drive; Lake Forest, CA 92630, USA; joan_stevens@agilent.com
[2]Agilent Technologies, Santa Clara, CA

High throughput food laboratories using QuEChERS for multi-residue analysis in a variety of matrices face significant challenges since not all matrices will yield the same level of co-extractives. Nonvolatile co-extractives can contaminate the system and limit sample throughput. One approach is to replace dispersive SPE with a SPE cartridge containing more sorbent to increase capacity; however

this requires a conditioning step, vacuum/positive pressure system, and evaporation of eluted volume - all of which are avoided by utilizing QuEChERS methodology. We have implemented a simple strategy when using a "just enough" sample purification technique such as QuEChERS in order to maintain GC/MS/MS performance. This involves a second dispersive step in series in order to streamline multi-matrix sample preparation. Modifying QuEChERS by implementing a second dispersive SPE tube does not increase the price per sample substantially and is still significantly less expensive overall (> 30%) than cartridge SPE.

Consecutive injections of post-spiked 2xLOQ winter squash were made over approximately 36 hours. New GC columns and liners were used for each sequence of 75 injections. The MS source had over 740 injections by the end of the 2x d-SPE sequence. Responses are normalized to the first data point. Highest %RSDs were obtained for carbophenothion (8.5), parathion ethyl (8.4) and phorate (7.0) in the 1x d-SPE sample. Overall averages of %RSDs for the 1x d-SPE and 2x d-SPE samples over 75 injections were 3.4 and 2.6, respectively.

P21 Chromatographic Results for Several Structural Isomers Obtained from Sub 2μ vs. Conventional 3μ C18 Columns

Eugene Chang, Shannon Lane, Mihai Kiss, Ajayi Olusegun and Irene Cassias, *Pacific Regional Laboratory, Southwest, FDA, Irvine, C;* Olu.Ajayi@fda.hhs.gov

Tandem LCMS is a powerful tool for compound separation and detection. However, it has limitations in separating structural isomers solely based on precursor and fragment ion transitions. Therefore, it is imperative to establish a good column separation condition to obtain acceptable quantification and identification of target compounds of structural isomers.

We compared chromatographic results for several structural isomers obtained from Sub 2μ vs. conventional 3μ C18 columns. Sub 2μ columns effectively achieved the baseline separation while 3μ columns could not. The examples include the pesticide and industry chemicals found in food matrices. These results also show a significant improvement in column technologies in recent years with better separation capabilities.

P22 QuEChERS in the Rye: Cleanup Comparison on QuEChERS extracts for Grains

Jessica Netzer, Tom Dobbs, Jeff Wiseman, Jennifer Salmons

J2 Scientific, 1901 Pennsylvania Drive, Suite C; Columbia MO 65202 USA; jnetzer@j2scientific.com

In pesticide analysis, sample clean-up is critical to guarantee accurate and precise results while minimizing the damage done to analytical instrumentation. Many factors are involved in choosing the right sample technique for any analytical method including the cost and potential hazards of reagents used, the time it takes to process each individual sample, and the number of manual steps requiring technician intervention during the process. The QuEChERS extraction provides fast and inexpensive cleanup for sample extracts across a variety of analyte classes and matrices but is not always effective in samples with a high fat content or other interferences that cannot be removed by dispersive solid phase extraction (dSPE). Traditional SPE and gel permeation chromatography (GPC) clean-up methods can require more time and/or solvent to complete analysis, but better cleanup results in higher recoveries, increased precision, and decreased stress on sensitive analytical equipment. Automating these traditional methods provides additional savings that can make SPE or GPC clean-up an alternative to dSPE in terms of time and expense per sample while providing a more thorough sample clean-up.

This presentation compares dSPE cleanup after QuEChERS extraction to traditional SPE and GPC clean-up methods for grains, examining the advantages and disadvantages of each technique for pesticide analysis.

P23 Maximizing Lipid Load With 2-Dimensional GPC Cleanup

Jessica Netzer, Tom Dobbs, Jeff Wiseman, Jennifer Salmons

J2 Scientific, 1901 Pennsylvania Drive, Suite C; Columbia MO 65202 USA; jnetzer@j2scientific.com

The maximum recommended lipid load for a 5mL injection onto the traditional GPC clean-up column described in EPA Method 3640A is 1 gram. This limitation sometimes creates the need to perform GPC cleanup on multiple aliquots of the same sample in order to remove the lipid while cleaning up adequate sample to reach desired limits of detection. Multiple fractions make a time- and solvent-intensive cleanup method like GPC more costly.

The PrepLinc 2D GPC feature allows the user to control the GPC Dump, Collect and Wash steps through two columns in series, increasing the lipid capacity. A high-lipid sample can be injected onto the GPC with the first Dump to waste passing only through the first column. When the majority of the lipid has been removed from the sample, the second GPC column is switched inline to complete the separation. The lipid loaded onto the second column is reduced by the first column, maximizing separation in a single

sample injection. Traditional GPC cleanup methods may require multiple GPC runs to achieve the same lipid removal for the large samples required to achieve very low limits of detection. This study demonstrates the effectiveness of 2D GPC on high-lipid samples.

P24 Combining Dispersive Solid Phase Extraction Cleanup and Particulate Filtration for QuEChERS Extracts in One Simple Step

Jack Cochran, Michelle Misselwitz, Julie Kowalski, and Sharon Lupo, **Restek Corporation**, 110 Benner Circle, Bellefonte, PA, 16823, USA; jack.cochran@restek.com

Dispersive solid phase extraction is an important part of the QuEChERS sample preparation approach, as it allows a fast cleanup of extracts by shaking with loose sorbent material. Unfortunately, half, or even more, of the sample is lost to the procedure. This marginalizes a concentration step for improving GC detectability, and eliminates the possibility of using more sorbent to increase cleanup capacity. In addition, samples may still need filtration prior to GC or LC analysis if they contain particulate matter.

We used an innovative, eight-position, vacuum-driven system that offers a convenient, high-throughput alternative to syringe-tip filters and allows filtration directly into autosampler vials. The dSPE sorbent and MgSO4 for QuEChERS cleanup can be poured directly into filter bodies from conventional pre-measured dSPE tubes. After vacuum is applied, sample volume recovery is essentially 100%, even if twice as much sorbent is used, and the cleaned samples are simultaneously filtered with 0.20 or 0.45µm PTFE filters, making them ready for LC and GC analysis.

P25 Feasibility of dilute-and-shoot LC/MS/MS, solvent-only calibration and multiple food types for multi-residue pesticide analysis: lazy chemists and old instruments

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

Increased selectivity and better sensitivity of LC/MS/MS have impacted how multiresidue methods are performed sometimes by decreasing the need for rigorous sample preparation. However, this technique is susceptible to matrix effects causing poor data quality and quantification to be difficult. Often instrument manufactures tout the dilute-and-shoot technique which greatly decreases processing time as no sample cleanup is performed but it also often requires the sensitivity of the newest instrumentation. Using a robust but older LC/MS/MS with sensitivity limitations, we investigated two aspects of multiresidue methods, sample preparation and calibration strategies, to determine the best procedures considering both data quality and time/cost. A variety of food types and two sample preparation methods, dilute-and-shoot and QuEChERS methods were performed and experimentally determined matrix effects were compared. Calibration methods were evaluated by determining recovery values using both solvent-only calibration and matrix-matched calibration. This allows assessment of sample preparation/calibration method combinations. We determined that with the easiest commodities, the dilute-and-shoot method and solvent-only calibration gave acceptable recovery values. However, for other commodities either a matrix-matched curve or cleanup was needed to obtain good recovery values. The high carbohydrate and citrus commodities proved to be too difficult. In almost every case, matrix-matched calibration provided improvement.

P26 Use of New Spherical Materials in a Dual-Layer SPE Tube for the Analysis of Pesticide Residues in Spinach.

Olga Shimelis, Katherine K. Stenerson, Ken Espenschied, Michael Halpenny, Ed Mauney, **Sigma-Aldrich/Supelco,** 595 Harrison Road, Bellefonte, PA 16823, USA; katherine.stenerson@sial.com

A new dual-layer carbon/NH2 SPE tube was evaluated for the cleanup of spinach extracts in the analysis of pesticides. The carbon material has a strong affinity for planar molecules, and can remove pigments and sterols that are commonly present in many food and natural products. The aminopropyl (NH2) sorbent functions to remove organic acids (including fatty acids), and will also retain some polar pigments and sugars. Both the new carbon and NH2 materials are spherical, allowing for improved and more consistent flow behavior between tubes. The improved flow characteristics of these materials allow faster gravity flow of the SPE tubes, eliminating the need for a vacuum manifold to process samples. In this study, we evaluated the new materials in a dual-layer tube, and compared them with conventional granular materials for color removal and pesticide recovery. The pesticides chosen for the study covered a variety of groups (organochlorine, organophosphorus, pyrethroid) and included polar and non-polar compounds. The new spherical materials were found to be highly effective at removing the chlorophyll present in the spinach extracts as well as yielding better recovery for a majority of the pesticides studied.

P27 Analysis of Pesticides in Fruits and Vegetables using Z-Sep and Z-Sep+ SPE Sorbents as Part of QuEChERS Approach to Sample Cleanup

Olga Shimelis, Katherine K. Stenerson, Emily Barrey, Michael Halpenny, Michael Ye, **Sigma-Aldrich/Supelco,** 595 Harrison Road, Bellefonte, PA 16823, USA; olga.shimelis@sial.com

QuEChERS, an acronym that stands for “quick, easy, cheap, effective, rugged, and safe” is a well-accepted method for the extraction and cleanup of a variety of food matrices in the analysis of pesticide residues. The method employs dispersive SPE for extraction by using salts and/or buffers to partition analytes into organic solvent, followed by a cleanup step using a separate sorbent. Common sorbents for cleanup include C18, primary secondary amine (PSA), and graphitized carbon black (GCB). In this work, two new cleanup sorbents, Z-Sep and Z-Sep +, were evaluated. Both materials are based on zirconia chemistry, and exhibit some degree of ion exchange with acidic character. Z-Sep was evaluated for the cleanup of orange and olive extracts, followed by LC-MS analysis of pesticides. The mix of pesticides included some acidic compounds, which were found not to retain on the Z-Sep sorbent. It was also found that Z-Sep combined with C-18 removed more pigment than PSA, and for both matrices, Z-Sep/C18 exhibited better recovery for several pesticides then both PSA and PSA/C18 mix.

Z-Sep+ has additional hydrophobic functionality on its surface, giving it the ability to remove fatty components. It was evaluated for the cleanup of avocado, a fat-containing matrix, followed by GC-MS analysis of hydrophobic and polar pesticides. For extract cleanup, Z-Sep+ was compared to PSA/C-18 and found to retain more total matrix, produce less interferences, and yield better pesticide recovery. In addition to Z-Sep+ cleanup, modifications to the extraction method to further increase pesticide recovery were also investigated.

P28 Pesticide Analysis Using QuEChERS Extraction: A Comparison of Manual and Automated Approaches

Tyler Trent, Tom Hartlein, Ed Price; **Teledyne Tekmar,** 4736 Socialville Foster Road; Mason, Ohio 45040, USA; ttrent@teledyne.com

QuEChERS is a Quick-Easy-Cheap-Effective-Rugged-Safe extraction method that has been developed for the determination of pesticide residues in agricultural commodities. QuEChERS itself has evolved from the original unbuffer method to a pair of multi-laboratory validated methods using citrate buffering (EN 15662:2008) or acetate buffering (AOAC Official Method 2007.01). While originally developed for plant matrices, the technique has since been adapted to be used in several applications and in many additional matrices. The rise in popularity of the technique and the increase in sample testing loads have lead to automation as a possible solution to increase productivity. The method, as mentioned, is a simple and fast extraction when compared to traditional semi volatile extraction techniques. However, it still requires dedicated manpower, sample prep equipment, manual spiking, manual pipetting, and a wide range of reagents and consumables. The analytical and practical challenges of automating this technique will be discussed and a comparison of extraction recoveries achieved both manually and by automation will be evaluated. Data will be presented that justifies automation as a possible solution to increase sample throughput, reduce costs, and improve the accuracy and precision of the extraction.

P29 Extraction of Spices using the SpeedExtractor E-916 for the Determination of Pesticides

[1]Mullis B, [1]Sander M, [1]Feifel S, [2]Cruzer C, [2]Schurmann A, [3]Maier U et al.

[1]BUCHI Labortechnik AG, Meierseggstrasse 40, 9230 Flawil, Switzerland; feifel.s@buchi.com; [2]Kantonales Labor Zurich, Switzerland; [3]Bachema AG, Ruttistrasse 22, CH 8952 Schlieren, Switzerland

Organochlorine pesticides (OCP) can cause severe health problems, are persistent and tend to bio-accumulate. Therefore the use of some OCP is banned by the Stockholm convention.

The efficacy of pressurized solvent extraction with BUCHI's SpeedExtractor was examined for the extraction of OCP and other pesticides in chilli and paprika spice samples. After reduction of the volume with Syncore® Analyst the extracts were cleaned up by GPC and analyzed by GC-MS/MS. In addition the extraction of OCP from certified reference soil material (CRM) was carried out. After the concentrated extracts had been cleaned up on Florisil the eluates were concentrated again and quantified by GC-ECD.

The measurements presented show close correlation with the target values of the certificated samples and to those results conducted by Accelerated Solvent Extraction ASE® 200. When not only the time that elapses until the result is generated but rather the throughput is of importance, SpeedExtractor opens new dimensions. SpeedExtractor delivers a throughput of up to six times that achieved when using the conventional sequential format. For the above mentioned applications six samples in parallel were extracted in 60 minutes and even less time.

We greatly acknowledge Mr. Claudio Cruzer and Dr. Andreas Schurmann of the Kantonales Labor Zurich, Switzerland and Mr. Urs Maier and his team of Bachema AG, Schlieren, Switzerland for their measurements and support in developing these applications.

P30 Direct Sample Analysis: A new ionization technology for rapid pesticide screening & streamlined analysis

Courtney Phillips, Avinash Dalmia, and Sharanya Reddy; **PerkinElmer**, 710 Bridgeport Avenue, Shelton, CT, USA; Courtney.phillips@perkinelmer.com

The AxIon DSA system is a breakthrough approach to sample introduction that eliminates the need for front-end GC or LC separation, and allows samples to be directly ionized at the entrance to a mass spectrometer. By replacing traditional lengthy sample preparation and chromatographic methods, the AxIon DSA/TOF system is a unique tool for streamlining and simplifying laboratory workflows. The high resolution, sensitivity, mass accuracy and wide dynamic range of the Time-of-Flight mass spectrometer allows for dependable results in complicated matrices, and allows for simplified method development compared to triple-quad technologies. This novel technique has been demonstrated to screen for pesticides and residues directly from the surface of fruits and solid food products, from drops of water and juices, and more applications are being investigated each day. The patented atmospheric pressure chemical ionization design prevents ion suppression and eliminates metastable ions for clearer, more definitive data.

P31 A Simple Method for Determination of Fungicides and Metabolites in Orange and Apple Juices using Polymeric SPE and LC/MS/MS

Irina Dioumaeva, **Agilent Technologies, Inc.** 25200 Commerecntre Drive, Lake Forest, CA 92630, USA; irina.dioumaeva@agilent.com

In January 2012 the US FDA announced that it began sampling import shipments of orange juice and will deny entry to shipments testing positive for carbendazim. Carbendazim is not registered for use in the US, but it is legally used in many other countries exporting their fruit and juice to the US. We present an analytical method for determination of carbendazim and three other fungicide residues in orange and apple juice with easy sample preparation and LC/MS/MS detection. Developed for the most important systemic fungicides, carbendazim, thiabendazole, and imazalil, this method also includes imazalil metabolite, alpha-(2,4-dichlorophenyl)-1H-imidazole-1-ethanol, and can be easily expanded to incorporate a broader range of pesticides. A quick and easy solid phase extraction procedure provides clean extracts that without any preconcentration allow for detection of analytes at concentrations below 2 ppb. Quantification was performed using triphenyl phosphate as an internal standard. Excellent accuracy (% recovery) and precision (% CV) data were obtained for three concentration levels: 10, 50 and 250 ppb of each analyte.

P32 MULTIRESIDUE METHOD FOR DETERMINATION OF EIGHT NEW GENERATION FUNGICIDES IN GRAPES USING GC- µECD

Vilca.F.Z [(1)]; Valdemar. L.T [(1)]; J. Sanz-Asensio[(2)] & M.T. Martínez-Soria[(2)]

[(1)] Center of Nuclear Energy in Agriculture CENA. University of Sao Paulo. Av. Centenario 303 CEP 13400-970, PO Box 96. Piracicaba Sao Paulo (BRASIL); [(2)] Department of Chemistry. University of La Rioja. Madre de Dios, 51. 26006 Logroño (Spain); franz-cena-usp@hotmail.com

A multiresidue method was developed and validated for the determination of eigth new generation fungicides (kresoxim, fenhexamid, cyazofamid, benthiavalicarb, metrafenone, boscalid, piraclostrobin, dimethomorph) and "tetradifon" used as internal standard in grapes using a GC- µECD system; the method involve extraction with 10 mL of acetonitrile (HAc 1.0%) 4 g of anhydrous $MgSO_4$ and 1 g of NaCl, hand-shaken vigorously for 1 min, and centrifuged for 5 min at 5000 rpm. For the clean-up step an aliquot of 5 ml of the supernatant was transfer to another tube with 125 mg of PSA and 750 mg of magnesium sulphate, hand-shaken vigorously for 1 min and centrifuged at 5000 rpm for 5 min. Following centrifugation, the supernatant was placed into a vial and analyzed by GC- µECD equipment a HP-5 30 m x 0.32mm x 0.25 µm column was used. The method was validated using a grape sample from the variety "tempranillo" without pesticides residues used as blank, spiked at 0.2 and 2 mg kg^{-1}; the average recovery by the method varied from 70.2 to 123.5% with RSD% < 15%. The method showed linearity $r^2 \geq 0{,}99$ and the LOQ ranged from 0.05 to 0.2 mg kg^{-1}. In this work, a useful and fast method for identification and quantification of seven new generation fungicides was optimized using a GC-µECD system, validated by means of a targeted GC approach.

P33 Strategies for Rapid Screening and Quantitation of Pesticides by Direct Analysis in Real Time (DART) Ambient Ionization Mass Spectrometry

Elizabeth Crawford and Brian Musselman, **IonSense, Inc.**, 999 Broadway Suite 404; Saugus, MA 01906, USA; crawford@ionsense.com

Routine pesticide and fungicide use in the United States, as well as abroad warrants the need for analytical techniques that can rapidly screen and quantify residues in order to efficiently monitor produce products before reaching the consumer market. The allowable residue levels of chemical residues including pesticides and fungicides on produce are governed by the US EPA Code of Federal Regulations that sets tolerance levels based on the commodity. Of particular interest in the United States, carbendazim was found in imported orange juice and orange juice concentrates from BRASIL, where the use of that fungicide is legal and therefore rapid product screening would increase consumer safety in the US and worldwide. The Direct Analysis in Real Time (DART) ambient ionization technique offers the ability to rapidly screen the surface of produce for pesticides by a direct swabbing method and permits direct analysis of fruit juices in seconds. With automated sample introduction rapid screening and quantitative measurements can be completed using DART ionization coupled to high resolution accurate mass and triple quadrupole mass spectrometers.

Limits of detection using the DART ionization quantitative method coupled with Q Exactive and API 4000 QTRAP mass spectrometers were below 10 ppb for a 10 pesticide mixture detected in a variety of fruit juices, as well as for carbendazim directly analyzed from fruit juices. A number of the orange juices from the EU (5 juices), India (4 juices) and USA (3 juices) were screened for carbendazim and three juices all purchased in the EU tested positive for the fungicide at levels ranging from < 5 ppb up to 21 ppb.

P34 A Rapid Response for the High Throughput, Sensitive and Accurate Determination of Carbendazim from Juice and Juice Concentrate

[1,2]Frederic Ciner, [1,2]Ed Dabrea, **1 Jupiter Analytix, 2 Jupiter Environmental Labs,** 150 S. Old Dixie Highway; Jupiter, FL 34558 USA; Jupiterlabs.com

Sensitive, robust and high throughput modern liquid chromatography/tandem mass spectrometric (LC/MS/MS) assays were developed for the quantitative determination of Carbendazim in orange juice and frozen concentrate. Sample preparation involved both Quechers and dilute / shoot methodology. Separation was achieved on various analytical stationary phases at 50 C using a rapid gradient with MeOH/0.2% formic acid H2O/0.2% formic acid at a flow rate of 1.1 mL/min over 4 minutes. Detection was performed using electrospray ionization in positive ion Multiple Reaction Monitoring (MRM). The method displayed linearity and reproducibility with a signal to noise of greater than 50/1 at 100 PPT

P35 Determination of Carbendazim in Orange Juice by QuEChERS and LC/MS/MS Detection

Xiaoyan Wang and Michael J. Telepchak, **UCT**, 2731 Bartram Rd., Bristol, PA 19007, USA; xwang@unitedchem.com

Carbendazim is a broad-spectrum fungicide with planar structure that is widely used to control fungi and molds in crops and fruits. It is permitted to use in the European Union (EU) and many other countries, but is not registered for use in the USA. Coca-Cola reported the detection of carbendazim in its orange juice products in early 2012, thereafter US FDA set the rejection limit of carbendazim to be 0.01 mg/kg in imported orange juice products. In this study a simple, fast and effective method based on QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) extraction, dispersive solid phase extraction (dSPE) cleanup with primary secondary amine (PSA) and endcapped C18, followed by LC/MS/MS detection is developed to determine carbendazim in orange juice. Clean extract, excellent recoveries (96.6, 100.2, and 103.7 % for three fortified levels at 10, 50, and 250 ng/mL), good reproducibility (RSD < 5 %, n=4), linearity range of 2-500 ng/mL with R^2 of 0.9992, and limit of quantification (LOQ) of 1.4 ng/mL were achieved. Six orange juice samples were tested with this new developed method. Carbendazim was found to be 5.3 ng/mL in one of the six samples, the detected concentration was far below the EU's maximum allowable level of 200 ppb carbendazim in orange juice.

P36 Speciated Arsenic Contents of Some Foods

John Budin,[1] Walt Brandl;[2] and Emiliano Castellano;[3]

[1]**Mérieux NutriSciences Corporation**, 111 E. Wacker Dr. Ste 2300, Chicago, IL 60601 USA; john.budin@silliker.com; [2,3]Silliker, Inc.

The arsenic contents of some common foods has recently received media attention. While the analysis or contents of arsenic in foods is not a new subject, recent reports of arsenic in apple juice and baby food have raised concern from consumers. Arsenic can be found in many different forms where it can be categorized as organic and inorganic forms. From the perspective of relative toxicity, the organic forms of arsenic are reported to be considerably less toxic than the organic forms of arsenic. This poster will provide a review of arsenic toxicity and report the speciated arsenic contents of some foods.

P37 Rapid Detection of Pesticides in Fruit Juice Without Sample Preparation Using High Resolution Chromatography and Highly Sensitive Tandem MS

Dimple Shah[1], Jinchuan Yang[1], Gordon Fujimoto[2], Lauren Mullin[1], Jennifer Burgess[1], & Ken Rosnack[1]

[1] **Waters Corporation**, 34 Maple Street, Milford, MA, 01757, USA; ken_rosnack@waters.com; [2] Waters Corporation, Beverly, MA

Pesticide residues in fruit juices have always been an important food safety issue, especially taking into account the high consumption of juice by children. A recent report of the fungicide carbendazim in orange juice has drawn widespread public attention. Since carbendazim is not licensed for use on citrus fruits in the U.S., in January, the FDA began testing all shipments of orange juice from foreign sources.

Due to the low detection levels required by the regulatory bodies and the complexity of matrices in which the targeted analytes are present, identification and quantification of pesticides at low levels is a must.

Many published methods are capable of analyzing pesticides in fruit juice for regulatory purposes. However, sample preparation is required for these methods to minimize matrix interferences. With advances in LC-MS/MS technologies, a fast screening method using a simple 'dilute-and-shoot' approach was evaluated for multi-residue analysis of pesticides in orange juice. The combination of ultra performance LC with ultra sensitive MS facilitated the trace level detection of pesticides well below the legislative limit. This poster will focus on a pesticide screening method with 2 MRM transitions allowing for both screening and confirmatory analysis. The dilute and shoot method was shown to provide excellent repeatability for more than 150 injections of orange juice. An incurred carbendazim residue was quickly and easily detected and quantified well below the reporting level and confirmed using ion ratios.

P38 Fungicides in Fruit Juice: Sample Preparation and Analytical Strategies for Multiresidue Screening, Targeted, and Confirmatory Analysis

Michael S. Young and Kim vanTran, Waters Corporation, 34 Maple Street, Milford, MA 01757, USA; Michael_S_Young@waters.com

Analytical screening methods are performed to identify and quantify as many residues as possible using a single and simple sample preparation method. Depending on the selectivity of the screening method and the sensitivity of the analytical instruments available, a targeted or confirmatory analysis may be required for residues initially detected with the screening method. Recently, a non-registered fungicide residue, carbendazim, was detected in orange juice imported into the USA. In this presentation we will discuss sample preparation strategies for determination of multiresidue fungicides, including carbendazim, in fruit matrix. We will focus on the requirements for an initial screening method using a standard screening protocol (QuEChERS) and LC-MS/MS with low ppb limits of detection. We will then discuss SPE and other sample preparation strategies for targeted confirmatory analysis of carbendazim and other fungicides using LC/MS (with sub- ppb detection limits) and LC/UV (with low ppb detection limits). The methodologies will be demonstrated with incurred orange samples.

P39 Inter-laboratory Validation of a Fast and Sensitive UHPLC-MS/MS Method with Fast Polarity Switching for the Analysis of Lipophilic Shellfish Toxins

Jerry Zweigenbaum,[1] Thomas Glauner,[2] Ana Brana-Magdalena,[3] José M. Leao,[4] Begoña Ben-Gigirey,[3] Ana Gago-Martínez;[3,4]

[1]**Agilent Technologies**, 2850 Centerville Road, Wilmington, DE 19808, USA; j_zweigenbaum@agilent.com; [2]Agilent Technologies Sales & Services GmbH & Co. KG, Waldbronn, Germany; [3]European Union Reference Laboratory for Marine Biotoxins (EURLMB), Vigo, Spain; [4]Department of Analytical and Food Chemistry, University of Vigo, Vigo, Spain

Recently EU Legislation replaced the mouse bioassay method for lipophilic shellfish toxin testing with an LC-MS/MS method. There have been several reasons, among them, lower false positive and negative rates as well as better precision and accuracy of the chemical assay. This has been shown in the interlaboratory validation study coordinated by the EURLMB, and in some other independent interlaboratory validations carried out in the EU using different LC-MS/MS methodologies. Whereas two different HPLC conditions have been used, common for both methods is that the determination of all regulated lipophilic shellfish toxins requires acquisition in positive and negative electrospray ionization (ESI) to gain the best possible sensitivity. However, there is concern that throughput for the LC-MS/MS method is not sufficient for routine use. In this work we show the results of an inter-lab validation for a fast UHPLC-MS/MS method working under acidic conditions acquired in 3 labs and 4 different UHPLC-MS/MS instruments. Twelve lipophilic marine toxins were acquired in fast polarity switching using Dynamic MRM for optimizing the duty cycle of the MS. Azaspiracids and pectenotoxins were analyzed in positive mode and yessotoxins were analyzed in negative mode. The okadaic acid group was analyzed in both positive and negative ionization and results for both ionization modes were compared. The validation

was done using the experimental design and samples for an interlaboratory validation study over a 3-day period based on the EU harmonized SOP published by the EURLMB.

P40 Development and Validation of a Method for Stilbene Residues in Fish and Fish Products

Ryan Gibbs, Hanna Smith, Cory Murphy, Ross Potter, Lynn Watson and James D. MacNeil, **Canadian Food Inspection Agency**, 1992 Agency Drive, Dartmouth, NS, CANADA; lynn.watson@inspection.gc.ca

The stilbenes are a class of synthetic, non-steroidal growth promoters which have been banned for use in food producing animals in Canada, the US and the EU. Their use is suspected for the treatment of aquatic mould infections in fish. A simple, rapid, sensitive analytical method was developed and validated to quantitate and confirm the presence of diethylstilbestrol, dienestrol and hexestrol in fish by ultra performance liquid chromatotography – tandem mass spectrometry. The developed method includes an acetonitrile extraction followed by a hexane wash and SPE cleanup (C18) with quantitation using internal standards. The validated method has a linear range from 0-40 ng/g in target representative species, with LOD from 0.3 – 0.7 ng/g for tilapia tissue and from 0.4-1.4 ng/g for salmon, depending on the analyte. Recoveries ranged from 88 to 111% in tilapia and 99-144% in salmon for the analytes of interest. Method repeatability was determined to range from 6-15% RSD for tilapia and 4-32% RSD for salmon. Instrument repeatability ranged from 2-4% RSD for tilapia and 2-8% RSD for salmon. Intermediate precision ranged from 4 to 16% RSD for tilapia and 7-42% for salmon with Horrat values within typical limits of within-laboratory acceptability for all analytes in the matrices studied. CCα ranged from 0.2- 0.7 ng/g for tilapia and 0.4-1.4 ng/g for salmon. CCß ranged from 0.3-0.8 ng/g for tilapia and 0.4 and 1.5 ng/g for salmon.

P41 Simultaneous determination of antibiotic residues in water from fish farm cage net by LC-MS/MS

Sérgio H. Monteiro, Graziela C. R. Moura Andrade, Alyne S. Sousa, Jeane G. Francisco, Rafael G. Botelho, Franz Z. Vilca and Valdemar L. Tornisielo; **Universidade de São Paulo,** Centro de Energia Nuclear na Agricultura, Av. Centenário, 303; Piracicaba, SP 13416-903, **BRASIL**; 55 19 34294764, shmonteiro@cena.usp.br

Aquaculture production has notably increased in the last years in BRASIL. Many classes of antibiotics are commonly used in aquaculture worldwide to treat infections caused by a variety of bacterial pathogens in fish and as growth promoter. A method was optimized and validated for simultaneous monitoring of five drugs of two different antibiotic classes in surface water. Oxytetracycline, tetracycline, sulfametoxin, sulfatiazol and sulfamethazine were chosen as target compounds because they were selected for monitoring in BRASILian Residue Control in Fish, realized by MAPA (Ministry of Agriculture, Livestock and Food Supply) and have pattern of high consumption. The method is based on SPE with hydrophilic-lipophilic-balanced reversed-phase sorbent without additional sample clean up stage followed by liquid chromatography – electrospray tandem mass spectrometry (LC–MS/MS) in positive mode. The limits of quantification (LOQs) were lower than 7 ng/L for all compounds. The fortification studies with 7 replicates at the levels of 25 ng/L and 50 ng/L showed recovery ranged from 45 to 82% and repeatability expressed as relative standard deviations (RSD) were lower than 24%. Therefore, the LC–MS/MS method can be successfully applied for the quantitative determination of antibiotics in surface water from fish farm that utilize cage net.

P42 Imported Catfish and Food Safety

Karl Bell,[1] Cindy Foster,[2] Ashely Meredith,[2] Ashli Brown,[1] Jose Rodriguez,[2]and Darrell Sparks;[1]

[1]Mississippi State University, Department of Biochemistry, Molecular Biology, Entomology, and Plant Pathology; Mississippi State, MS 39762, USA; [2] **Mississippi State Chemical Laboratory**, 1145 Hand Lab 310 Presidents Circle; Mississippi State, MS 39762, USA; CFoster@mscl.msstate.edu

Channel Catfish (*Ictalarus puncatus*) is the fifth largest "crop" in Mississippi generating 217,774,000 dollars of revenue in 2011. Influx of fish species such as Basa (*Pangasius bocourti*) and Tra (*Pangasianodon hypophthalmus*) from foreign markets has a large economic impact and also poses certain health risks. Intentional misbranding of species, presence of antibiotics, and pathogenic microorganisms such as *Salmonella* sp. are the main threats to public health. To help curb these threats, several studies have been conducted. First, Fatty Acid Methyl Esters (FAMEs) derivatized from fish fillet lipid extracts were analyzed via gas chromatography to generate lipid profiles unique to each species of fish. Percent area of Myristic, Palmitic, Stearic, and Linoleic acid methyl esters were found to be significantly different among Catfish and Tra fillets. Next, a method to quantitatively determine nitrofuran (a class of antibiotics) metabolites via triple quadrupole LC-MS/MS was developed. Aminohydantoin, 3-amino-5-mopholinomethyl-2-oxazolidinone, 3-amino-2-oxazolidinone, and Semicarbazide were chromatographically separated and detected at concentrations as low as five parts per billion. Recoveries of these compounds varied from 80-110 percent. In tandem, these methodologies can be used to rapidly and correctly identify imported fish fillets and products contaminated with these agents.

P43 Detection of the Illegal Use of Trifluralin in Shrimp Farming

Danny Chan,[1] Richard Fussell,[1] Mike Hetmanski,[1] Jack F. Kay,[2] Andrew Grant,[3] David Verner – Jeffreys[4] and M. Sharman;[1]

[1]**Food and Environment Research Agency**, Sand Hutton, York, YO41 1LZ , UK; richard.fussell@fera.gsi.gov.uk; [2]Veterinary Medicines Directorate, Addlestone, UK; [3]VetAqua(Scotland), Muir of Ord, UK; [4]Cefas Weymouth Laboratory, Weymouth, UK;

Recent intelligence reports suggest that trifluralin, a herbicide and suspected carcinogen (now banned in the EU) is being used in the production of shrimps in certain third countries. Residues of trifluralin parent at over 1 µg/kg in Vietnamese shrimps exported to Japan were reported in 2010. The high number of transformation products (metabolites and degradation products) of trifluralin reported in the literature suggests that the use of trifluralin parent to monitor and detect misuse may be questionable. In an attempt

to identify the most suitable marker compound, juvenile Pacific shrimp (*Litopenaeus vannamei*) were exposed to trifluralin (at two different concentrations) for 72 hours under controlled conditions. Samples of shrimp collected at several time points (up to 48 days after treatment) were analysed along with samples of water and biota. Single-quadrupole GC-MS was used for the quantitative analysis of trifluralin, whilst LC-qTOF-MS was used in combination with profiling and metabolite ID software to screen for possible transformation products. Trifluralin and two possible transformation products giving a relatively high response by LC-qToF-MS were detected.

Information on the experimental design, analytical methods and emerging results will be presented.

P44 Decreasing the Impact of Aflatoxin in Mississippi

Ashli Brown,[1,2] Darrell Sparks,[1,2] Cedric Reid,[1] Andrew McDaniel,[2] Olga Pechanova,[2] Jose Rodriguez,[2] Paul Williams[3]

[1]**Mississippi State University, Department of Biochemistry, Molecular Biology, Entomology, and Plant Pathology**, 32 Creelman St.; Mississippi State, MS 39762, USA; [2]**Mississippi State Chemical Laboratory**, 310 Presidents Cr.; Mississippi State, MS 39759, USA; abrown@bch.msstate.edu; [3]USDA Agricultural Research Station, Corn Host Resistance Unit, Mississippi State, MS

Agriculture is Mississippi's leading industry generating $6.3 billion in revenue and employing approximately 29 % of the state's workforce. Last year MSCL used ELISA to test 752 corn-crop and 287 corn-based feed samples for total aflatoxin contamination. Aflatoxin concentrations ranged from 0-7800 ppb for corn and 0-190 ppb for corn-based feed samples. Last year the corn industry estimated a loss of $192 million. A breakthrough in mycotoxin detection or elimination would provide the American farmer with the tools to protect investments and remain competitive in the global market. We are using a systems biology approach to better understand the complex host-pathogen interaction, and develop a conceptual model to predict the occurrence of emerging toxins. Partnering with Agilent Technologies we have developed a LC-MSMS method to monitor individual aflatoxins. We have analyzed 410 corn samples from 6 counties, with soil pH ranging from 5.88-6.99, and aflatoxin concentrations of N.D.- 100 ppb. Coupling this data to weather conditions and agronomical practices we hope to elucidate an aflatoxin outbreak model. Using SPME-GC-MS we have identified a volatile analyte that can be linked to aflatoxin production. We have constructed a 2D proteome reference map of the aflatoxigenic fungus *A. flavus*. Using 2-DE and MALDI-TOF-MSMS, we identified 536 mycelial proteins, which the majority of were functionally annotated as related to cellular metabolic processes, biosynthesis, and enzymes from aflatoxin synthesis pathway. Additionally, using DGE and qRT-PCR we have identified several genes that show an increase in expression during aflatoxin reduction.

P45 Improved Method for Determination of 3-chloro-p-toluidine hydrochloride (DRC-1339) Residues in Avian Tissues by GC/MS/MS

Steven F. Volker, Randal S. Stahl, **National Wildlife Research Center**, 4101 Laporte Avenue, Fort Collins, CO 80521, USA; Steven.F.Volker@aphis.usda.gov

An improved method for DRC-1339 (3-chloro-4-methylaniline hydrochloride) residue analysis in avian tissues using GC/MS/MS operated in multiple reaction monitoring (MRM) mode was developed. DRC-1339 is the only registered avicide for use in the United States for controlling blackbirds, starlings, pigeons, gulls, magpies and ravens that damage agricultural crops, personal property or prey upon federally-designated threatened or endangered species. Following addition of the surrogate, hexadeuterated DRC-1339, homogenized tissues were extracted with an acetonitrile/HCl solution, neutralized, and extracted into hexane. Extracts were then cleaned-up with Si SPE cartridges, eluted with n-butyl acetate, and chromatographed on a DB-5MS column configured with column backflushing. In comparison to the original GC/MSD method, run times were shortened by 50%, accuracy was increased, and both sensitivity and selectivity were improved by monitoring multiple transitions for DRC-1339.

P46 Analysis of Stilbene Residues in Aqucultured Finfish using LC-MS/MS

Wendy C. Andersen, Jack J. Lohne, Sherri B. Turnipseed, and Mark R. Madson, **US Food and Drug Administration, Animal Drugs Research Center**, Denver Federal Center Bldg. 20, W. 6th Ave and Kipling Blvd; Denver, CO 80225-0087, USA; wendy.andersen@fda.hhs.gov

Diethylstibesterol (DES), dienesterol (DEN), and hexestrol (HEX) are non-steroidal synthethic estrogenic compounds belonging to the stilbene class of compounds. Prior to 1980, DES was commonly used both in human medicine for reproductive conditions and for livestock growth production and treatment. DES has since been found to be an endocrine disruptor that causes cancer and birth defects in humans. DES and other stilbene compounds have been banned from use in food animal production in the US, the EU, and in other countries. Reports in the literature indicate that DES has been studied both to increase growth and to induce male to female sex reversal in aquacultured fish. To determine the possibility of harmful stilbene residues being present in the food supply, a simple LC-MS/MS method was developed to detect residues of DES, DEN, and HEX parent compounds in aquacultured finfish. Homogenized fish tissue was extracted with acetonitrile and extracts were purified with silica solid phase extraction. The method was validated in catfish, salmon, tilapia, and trout muscle fortified at 0.5, 1.0, and 5.0 ng/g. Residues were positively identified by matching product ion ratios from 3 transitions. With internal standard correction (d^8-DES), residue recovery was generally in excess of 90 % with % RSD < 20 %. Method detection levels for each matrix were found to be below 0.5 ng/g.

P47 Sample Preparation for UPLC-MS/MS Determination of Aminoglycoside Antibiotics in Meat and Milk

Michael S. Young, Kim vanTran, Jeremy C. Shia, John Martin and Kenneth J. Fountain; **Waters Corporation,** 34 Maple Street, Milford, MA 02019 USA

Aminoglycosode antibiotics are commonly used in veterinary medicine and are used for treatment of animals grown for meat and milk production. This class of antibiotics presents significant challenges for residue analysis of tissue and milk. Unlike most

other antibiotic classes, these compounds are not amenable to extraction with acetonitrile or other organic solvent. Typically, the aminoglycosides are extracted from meat tissue using an aqueous buffer with trichloroacetic or perchloric acid added to precipitate protein and to inhibit protein binding of the analytes. Prior to LC/MS analysis it is desirable to remove the residual acids and any co-extracted interferences. This presentation will discuss SPE cleanup options prior to UPLC/MS determination of aminoglycosides in kidney tissue and milk. The aminoglycosides themselves display very limited reversed-phase retention. The amino functionality common to these analytes allows for retention on reversed-phase sorbents using ion-pairing reagents such as HFBA. Alternatively, ion-exhange sorbents can be employed. Among the SPE options discussed will be ion-pairing reversed-phase, weak and strong cation-exchange and hydrophilic interaction.

P48 Multi-Target Comparative Screening for Pesticides using High Resolution and Accurate Mass LC-MS/MS

Andre Schreiber[1], Axel Besa[2]

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

LC-MS/MS using Electrospray Ionization (ESI) is a powerful analytical tool for the analysis of a wide molecular weight range of polar, semi-volatile and thermally labile compounds. Especially triple quadrupole based mass analyzers are popular for targeted quantitation of hundreds of food contaminants in a single analysis because of their extra degree of selectivity and sensitivity when operated in Multiple Reaction Monitoring (MRM) mode.

Advancements in LC-MS/MS technology, including hybrid systems like triple quadruple linear ion trap (QTRAP®) and tandem quadrupole Time-of-Flight (TripleTOF™), now provide the ability to identify target analytes with enhanced confidence and to combine targeted and non-targeted screening on a routine basis.

Here we present residue results of using a novel approach of comparative multi-target screening. Fruit and vegetable samples were extracted using a generic QuEChERS extraction and generic reversed phase LC separation method. High resolution and accurate mass MS and MS/MS information was collected in a single run using information dependent acquisition on an AB SCIEX TripleTOF™ 4600 system. TOF-MS information was used to screen for targeted food contaminants. Automatic identification was based on retention time, accurate mass and isotopic pattern of the quasi-molecular ion. Quantitative information was achieved by performing a single concentration standard addition at the level of the Maximum Residue Level (MRL). For additional confidence in identification the accurate mass MS/MS spectra were searched against a mass spectral library. The molecular fingerprint saved into MS/MS spectra allowed to differentiate isomeric compounds and greatly reduced the number of potential false positive results.

P49 Identification of Pesticide Residues in Food Samples using LC-MS/MS and Non-Targeted Data Processing Strategies

Scott Kragerud[1], Andre Schreiber[2], Carmai Seto[2], Eva Duchoslav[2]

[1]**AB SCIEX**, 353 Hatch Dr, Foster City, CA, USA; Scott.Kragerud@absciex.com; [2]AB SCIEX, Concord, ON, Canada

Liquid Chromatography coupled to tandem Mass Spectrometry is a powerful analytical tool for the analysis of polar, semi-volatile, and thermally labile compounds of a wide molecular weight range, such as pesticides and other food residues. Mass analyzers based on triple quadrupole technology operated in Multiple Reaction Monitoring (MRM) mode deliver highly selective and sensitive quantitative results and are therefore well established for multi-target screening and quantitation of food contaminants.

However, the use of triple quadrupole based mass analyzers is limited to targeted screening and quantitation. But there is an increasing demand for retrospective and non-targeted data analysis. High resolution and accurate mass instruments are capable of performing targeted and non-targeted screening in a single LC-MS/MS run.

Here, a generic QuEChERS procedure was used to extract pesticides from a variety of fruit and vegetable samples. Extracts were subsequently analyzed by LC-MS/MS using a polar embedded C18 phase and an AB SCIEX TripleTOF™ 4600 system operated in high resolution accurate mass MS and MS/MS mode. Full scan MS data was explored to identify residues using non-targeted data processing workflows. Automatic sample-control-comparison was successfully used to detect pesticides in different food samples. Sample-control-comparison was found to be a quick and easy tool to identify residues. Different software tools to identify and characterize chemical structures based on MS and MS/MS information, including empirical formula calculation, automatic searching of the internet for possible structure identities, and MS/MS fragment ion prediction, were investigated.

P50 Screening of Unknown Pollutants in Water Sample Using High Resolution and Accurate Mass LC-MS/MS

YunYun Zou, André Schreiber, Robert Ellis, **AB SCIEX**, 71 Four Valley Drive, Concord, Ontario, Canada; Yunyun.zou@absciex.com

Fresh water quality closely impacts humans' life. There are a variety of pollutants present in fresh water, including pesticides, antibiotics, drugs of abuse, hormones, etc. It is necessary to accurately monitor their presence to assess the compounds effect on environment and life. It is challenge to analyze them together due to the diversity of chemical properties. Here we present a LC-MS/MS method for water sample with identification of unknown pollutants based on multi-class MS/MS library searching. High resolution accurate mass data improve the confidence of the results and reduce false positive response. In addition, the fast scan speed allows collecting the full scan TOF-MS data and TOF-MS/MS which can be revisited and explored for more non-targeted or unexpected compounds later. 21 Water samples were collected from rivers, creeks, lakes and local drinking waters in different cities and countries. Large volume direct injection is chosen in this method to achieve sufficient sensitivity. Full scan high resolution and MS data were used to identify the unknown pollutants in the sample and automatically triggered TOF-MS/MS spectra are used to further identification of targeted compounds with MS/MS spectral library searching. The MS/MS spectra of over 2400 compounds including drugs of abuse, pesticides, veterinary drugs, mycotoxins and their metabolites are in the library to provide a large amount

of database for library searching. A user-friendly data mining and processing software was used to do library searching and save time in processing the large amount of data.

P51 Finding Pesticides using an Agilent 7200 GC/Q-TOF and Unknowns Analysis Software

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

Most GC/MS pesticide residue methods are designed to look for a target list of compounds. A lab typically analyzes samples on a GC single quadrupole instrument in the selected ion monitoring (SIM) mode or on a GC triple quadrupole instrument in the multiple reaction monitoring (MRM) mode. In either case, there is a finite list of target compounds and, if a pesticide is not on that list, it will be missed with no possibility of retrospective analysis. This poster focuses on the use of an "unknowns analysis" program to search for non-target compounds after the target analysis is complete. If additional pesticides are found, they can be added to the target list for subsequent runs. The data were acquired on a high resolution, accurate mass Agilent 7890/7200 GC/Q-TOF operated in the TOF mode. In this mode, the full EI spectra are acquired throughout the run and are available for both target and non-target analysis.

P52 Ultra-high Performance Liquid Chromatography Coupled with Electrospray Ionization Quadrupole Time-of-Flight Mass Spectrometry for Simultaneous Screening of Multiple Pesticides in Scallion

Meiling Lu, Wenwen Wang, Shan Zhou; **WW Food Team China Segment, Chemical Analysis Group, Agilent Technologies,** No.3, Wangjing North Road, ChaoYang District; Beijing, 100102, China; shan_zhou@agilent.com

Pesticides in fruits, vegetables and other edible plants have long been the focus for food safety. Scallion, a widely used cuisine condiment in Asia, is noxious for its complicated interference during conventional analysis. Here we have developed an ultrahigh performance liquid chromatography (UHPLC) combined with electrospray ionization (ESI) quadrupole time-of-flight (QTOF) mass spectrometry (MS) method for simultaneous screening of multiple pesticides in scallion. The homogenized scallion sample was subjected to extraction by acetonitrile and purification using Agilent Bond Elut Carb/ NH_2. The resultant samples went through UHPLC-ESI-QTOF/MS analysis under a 30-min gradient elution with on-line MS calibration. It was found that all the pesticides (79) studied display good linear responses, with the linear regression coefficients (R^2) for the matrix-matched calibration curves all above 0.95 within the spiked concentration range (10-200 ng/mL), among which 82% of pesticides having R^2 of 0.99 or higher. At the lowest calibration level (10 ng/mL), corresponding to 1 ng/g pesticides spiked in the sample, the signal to noise ratio (S/N) for all pesticides is greater than 3, and 52% pesticides can give an S/N of 10 or higher. The recoveries for most pesticides are within 60-130% at the spiking level of 10 ng/g. It suggests that the UHPLC-ESI-QTOF/MS method developed here is sensitive and reliable, thus can be applied to screen multiple pesticides at the level of 1 ng/g, which is well aligned with the screening requirement for the strictest regulations such as EU regulations at the level of 10 ng/g.

P53 Analysis of Multiple Pesticide Residues in Scallion by Gas Chromatography Quadrupole Time of Flight–Mass Spectrometry (GC /Q-TOF)

Wenwen Wang, Shan Zhou; **Agilent Technologies (China) Co Ltd**, No.3, WangjingbeiLu, Chaoyang District; Beijing, 100102, China; shan_zhou@agilent.com

Scallion is not only a Chinese traditional food condiment, but also one of the important agricultural goods for export. Traditional analysis methods, such as gas chromatography or gas chromatography-single-quadrupole mass spectrometry, were susceptible to the strong interference of scallion matrix, resulting in inaccurate test results. This research describes method for analysis of 100 pesticide residues in scallion using the Agilent 7200 GC/Q-TOF. The 100 pesticides for this experiment are from Chinese regulation list. The homogenized scallion sample was subjected to extraction by acetonitrile and purification using Agilent Bond Elut Carb/ NH_2. Data were acquired using GC Q-TOF in scan mode and external standard was used for quantitative determination. Initially scan mode was applied to determine the retention time, then an accurate mass database of 100 pesticides was created, and two specific accurate mass ions were selected for each pesticide. The linear correlation was determined with the concentration ranging from 10 µg/L to 200 µg/L among which, majority of the linear correlation coefficients of the pesticides are higher than 0.990, with majority of pesticides be detected at the spiked level of 10 µg/L. With the same sample extraction and purification procedure, three pesticides were detected using triple quadrupole GC/MS/MS, and the same results were also obtained while using GC-QTOF method. It suggests that the GC /Q-TOF is highly sensitive, reliable, and can be applied to screen the 100 pesticides at low level in scallion.

P54 Exact Mass MS/MS Spectral Library for Screening Non-targeted Pesticides and Other Contaminants in Food and Environmental Samples using LC-QTOF-MS

Jerry Zweigenbaum,[1] Sebastian Broecker,[2] Fritz Pragst;[2]

[1] **Agilent Technologies**, 2850 Centerville Road, Wilmington, DE 19808, USA; j_zweigenbaum@agilent.com; [2] Charite University Hospital, Berlin, GERMANY

Liquid chromatography in combination with hybrid quadrupole time-of-flight mass spectrometry (LC-QTOF-MS) with use of a database and a CID spectral library is demonstrated to be one of the most efficient methods in screening for non-targeted compounds. In this study the advantages of an exact mass spectral library and the limitations of this technique for substance identification are investigated under practical conditions. Databases containing large lists of compounds of interest such as pesticides, pharmaceuticals, and poisons are very useful for screening with accurate mass measurement without standards. False positives and false negatives are major concerns for screening food and environmental samples. False negatives can occur from very low concentrations, matrix interferences and suppression, and poor or no ionization. These causes can be determined only using standards spiked into the sample matrix. False positives can occur from complex samples with many ions present. Using mass measurement even at better than 2 ppm accuracy, complex samples can produce many false positives using a large database search

only to make routine screening untenable. This study demonstrates the use of accurate mass MS/MS library searching in identifying true and false positives found by database search in surface waters and various food extracts. For example dodemorph was found by database search in surface water with 0.03 ppm error but its library spectrum was not that of dodemorph. In contrast carbendazim was found in the same sample with a 1.3 ppm error and was positively identified (but not confirmed) by library search.

P55 Differential Analysis of Organic and Non-Organic Honey for Pesticides and Pollutants by LC Time-of-Flight Mass Spectrometry

Joan Stevens,[1] Dawn Stickle,[2] Sue D'Antonio[3]

[1]**Agilent Technologies**, 25200 Commercentre Drive, Lake Forest, CA 92630, USA; joan_stevens@agilent.com; [2]Agilent Technologies; Minook, IL; [3]Agilent Technologies, Hudson, FL

In recent years, organic certified foods have grown in popularity due to their perceived potential to contain, for example, less pollutants and pesticides. Food must meet certain criteria established by an organic certification body in order to be certified organic. Certified organic honey, for example, must meet criteria, including but not limited to, the source of the nectar, the honey bees foraging area, bee management and the honey extraction processes. For honey, in particular, this requires considerable resource as it is hard to restrict bees' travel, making it possible for them to forage on non-organic pollen. For many beekeepers it is too difficult to adhere to these strict organic guidelines thus questioning the purpose of purchasing organic honey. Six different types of honey, from a mix of different regions and some certified organic (by different certifying bodies) were purchased. The honey samples were each treated using an identical QuEChERS extraction protocol. Each sample and a pesticide standard (containing approximately 200 common pesticides) were analyzed (n=6) using reversed phase liquid chromatography coupled to a time-of-flight mass spectrometer. The data was processed using an algorithm capable of reducing the data set into unique compounds found throughout the chromatographic run. The list of compounds was then filtered by a database and matched using exact mass. The results were then subjected to differential analysis to indicate relative similarities and differences between the different types of honey. Since matches will only be made on retention time and exact mass, the pesticide standard will be used as an additional sample to provide, if possible, additional confirmation for certain pesticides

P56 Novel Scientific Information System Evaluated for Semi-Quantitative High Throughput Screening of Pesticide Residues in Food

Gareth Cleland[1*], Mike McCullagh[2], Jennifer Burgess[1], Dimple Shah[1], Lauren Mullin[1], Sara Stead[2], and Ken Rosnack[1]

[1] **Waters Corporation**, 34 Maple Street, Milford, MA, 01757, USA.; ken_rosnack@waters.com; [2] Waters Corporation, Manchester, UK.

Pesticides control plant pests and improve crop production. Currently, more than 500 diverse pesticides are routinely used globally, with countries differing in regulations concerning licensing and maximum residue limits (MRLs). To protect consumer safety with increasing global trade, multi-analyte screening strategies capable of efficiently detecting residue violations are required. In recent years the use of accurate mass instruments has been at the forefront of food and environmental screening discussions: and this has also been reflected in the evolving CODEX guidelines that acknowledge this challenge and promote the use of accurate mass technology to address the challenges faced by industry.

In this pesticide screening study, the advantages of UPLC along with advances in TOF technology and novel informatics enabled full spectra acquisition and quantitation to be performed in complex matrices routinely at concentrations equivalent to 0.5 MRL. Over 100 pesticides were spiked into mandarin matrix and targeted against a database of 479 pesticides held within a scientific information system. Correlation coefficients of 0.99 have been illustrated, residuals obtained of <5%, and mass accuracy of <2ppm obtained. Processing data through this new scientific information system enabled confirmation of pesticide residues with confidence through the use of a Peak 3D detection algorithm where data is componentized, spectra are time aligned and cleaned to provide the best quality data available. Using this scientific information system to perform data acquisition, data processing and data management requirements together with access to an extensive scientific library provides a comprehensive workflow to screen, quantify, and confirm residues in a single solution.

P57 Utilization of Rapid LC-MS for Screening and Quantitative Analysis of Pesticides in Food Matrix using an Orbitrap platform

Cory Lytle[2], Michal Hauer[2], Charles Yang[1], Leo Wang[1], Jonathan Beck[1] and Dipankar Ghosh[1]

[1]Thermo Fisher Scientific, 355 RiverOaks Parkway, San Jose, CA 95134; [2]**Thermo Fisher Scientific**, 1400 North Pointe Parkway, West Palm Beach, FL; michael.hauer@thermofisher.com

Current methodologies for the quantitation of pesticides in food revolve around using triple quadrupole platforms and long run times. The method described here utilizes UHPLC with an Exactive Plus mass spectrometer using high resolution accurate mass. The Exactive Plus MS can resolve matrix from analyte in full MS mode and even in the most complex matrices with resolving power settings of up to 140,000 (FWHM). This work describes a method to quantitate 110 pesticides mixture together with high level of confirmation. A Hypersil Gold aQ C18 50x2.1mm 1.9u column was utilized with a run time of less than 8mins. A standard curve with 110 pesticides was spiked in green bell pepper solution ranging from 0.050 - 250 ng/mL and injected in triplicate and screening of green bell pepper samples for possible other unknown pesticides. The spectrometer was set to a resolving power of 70,000 (FWHM) in full MS mode to minimize matrix interference and all-ion-fragmentation spectra were collected. The data was then compared to current MS/MS library for confirmation, as well as calibration curves were generated for the individual compounds. Calibration lines for the compounds analyzed with $r^2 \geq 0.9930$, the LOD varied from 50 - 500pg/ml based on the individual compounds. Confirmation

of exact mass, isotopes, as well as the AIF produced were collected and compared, to limit the amount of "false positives" in the results. One main challenge in using a high resolution accurate mass system is data mining, with ExactFinder software the data processing becomes straight forward.

P58 Multi-residue Pesticide Analysis of Botanical Dietary Supplements using Gas Chromatography- Tandem Mass Spectrometry (GC-MS/MS) and GC-High Resolution-Time-of-Flight (GC-HRT)

Douglas G. Hayward[1], Jon W. Wong[1] Feng Shi[1,4], , Kai Zhang[1], Nathaniel S. Lee[2], Alex L. DiBenedetto[2], Mathew J. Hengel[3], David Alonzo[5], Joesph Binkley[5]

[1] U.S. Food and Drug Administration, Center for Food Safety and Applied Nutrition, 5100 Paint Branch Parkway, HFS-706, College Park, MD 20740-3835; douglas.hayward@fda.hhs.gov; [2] Joint Institute for Food Safety and Applied Nutrition, University of Maryland, 1122 Patapsco Building, College Park, MD 20742-6730; [3] IR-4 Laboratory, University of California, Department of Environmental Toxicology, One Shields Avenue, Davis, CA 95616; [4] Present affiliation: Givaudan Flavors Corporation, 1199 Edison Drive, Cincinnati, OH 45216; [5] LECO Corporation, 1850 Hilltop Rd. Saint Joseph, MI, 49085

Dietary supplements form an increasingly important part of Americans diet with growing economic and health impact yet broadly applicable multi-residue pesticide methods have not been evaluated for most supplements. A method for the analysis of 311 pesticides, isomers and pesticide metabolites in dried botanical dietary supplements has been developed and validated using 24 botanicals with GC-MS/MS. Orthogonal confirmation using GC-HRT-MS was done as part of the validation. Each botanical was prepared by adding acetonitrile:water to the dried botanical product follow by with magnesium sulfate and sodium chloride to extract. Clean-up employed SPE using a dual bed column consisting of graphitized carbon black and primary-secondary amine sorbent. Accuracy and precision were evaluated using fortification at 10, 25, 100 and 500 ng/g. LOQs using GC-MS/MS varied from 0.5 – >40 ng/g determined statistically using matrix matched standards. Four botanicals (black cohosh root, ginseng, hops and saw palmetto) were measured using two orthogonal GC/MS techniques. Geometric Mean LOQs for 311 pesticides in these botanicals was 11 ng/g using GC-MS/MS. Eleven incurred botanicals were measured, 5 hop and 6 ginseng sources. Quinoxyfen was measured in three sources of hops (100-620 ng/g) by both instrumental techniques. Quintozene its metabolites (pentachloroaniline, pentachlorobenzene, pentachloroanisole, pentachlorothioanisole) and contaminants (hexachlorobenzene, tecnazene), along with BHCs and DDT were measured in 5 of 6 ginseng sources using either instrumental technique. Azoxystrobin, diazinon and dimethomorph were measurable in a sixth ginseng source. The addition of an orthogonal MS technique reduced the rate of false negative pesticide identifications in complex botanicals such as ginseng and hops.

P59 Quick and Easy Screening and Quantitation of Pesticides in Food and Biological Matricies using Orbitrap Mass Spectrometer

Marcus Miller,[1] Leo Wang,[1] Guifeng Jiang,[1]; [1]**ThermoFisher Scientific**, 355 River Oaks Parkway, San Jose, CA, USA; marcus.miller@thermofisher.com

Pesticides screening and quantitation is important for monitoring in environmental and food safety applications. Analysis is complicated because of the wide range of chemicals that may be present in samples as well as complex matricies. Screening samples via LC/MS analysis is a common technique. To maximize throughput, short run times are required which leads to high demands on fast data collection. This necessitates sophisticated software to automatically evaluate and identify the data to maximize the efficiency and reduce workload for the scientist. Samples of 94 pesticides were analyzed in water, food extract and biological matricies using an orbitrap mass spectrometer. The ability to obtain high resolution and accurate mass data minimizes matrix effects; method development time is minimized or eliminated. Improved data acquisition rates mean that chromatographic run times can be kept short yet still allow for confirmations and quantitation of the pesticides. Workflow within the analysis software allows for easily collecting or importing the data. Parameterless peak detection quickly picks potential target analytes without the need for user intervention. All these factors contribute to a method that allows quick and easy screening and quantitation of pesticides in difficult matricies.

P60 Simultaneous Determination of Paraquat and Diquat in Environmental Water Samples by HPLC-MS/MS

Leo Jinyuan Wang,[1] Xiaodong Liu[2], Marcus Miller,[1,] and Guifeng Jiang[1] ; [1]**Thermo Fisher Scientific**, 355 River Oaks Parkway; San Jose, CA 95134, USA; marcus.miller@thermofisher.com; [2]Thermo Fisher Scientific; Sunnyvale, CA

Paraquat and diquat are widely used herbicides for both terrestrial and aquatic plants and their application poses concern for aquatic life and human health. Diquat is currently regulated by US EPA at 20 µg/L in drinking water. Analytical determination of both compounds has been challenging due to their high hydrophilicity. This study describes a high performance liquid chromatography tandem mass spectrometric method for high throughput, sensitive and selective quantitation of both compounds in environmental waters. Retention and separation were achieved on a specially designed prototype mixed mode column. Without using ion pairing reagent, sensitivity was significantly improved and detection limit was extended to sub-ppb levels. Environmental water samples were injected directly for online SPE cleanup using a weak anion exchange cartridge. Sufficient retention and improved chromatographic resolution were achieved with isocratic elution on the prototype mixed mode column featuring reverse phase, anion/cation exchange retention mechanisms. Analytical time was within 5 minutes per sample ensuring routine high throughput. Selected reaction monitoring was used for quantitation with isotope labeled internal standard to achieve quantitation accuracy. Lower limit of quantitation (LLOQ) was determined at 100 ng/L for both analytes. Precisions were within 10% at 500 ng/L and 4% at 50 µg/L. A good calibration range was achieved from 100 ng/L to 100 µg/L with coefficient of determination (r^2) greater than 0.999 for both analytes using 1/x weighting factor. Other performance parameters such as specificity, carry over, accuracy, matrix effect, and real sample analysis were also evaluated and will be presented.

P61 High Resolution LC-MS for Screening and Quantitative Analysis of Antibiotics in Drinking Water using an Orbitrap and Online Sample Preparation

Michael Hauer[2], Cory Lytle[2], Jonathan Beck[1] Charles Yang[1] and Dipankar Ghosh[1]

[1]Thermo Fisher Scientific, 355 RiverOaks Parkway, San Jose, CA 95134; [2]Thermo Fisher Scientific, 1400 North Pointe Parkway, West Palm Beach, FL; cory.lytle@thermofisher.com

Most current methodologies for the quantitation of antibiotics in drinking water revolve around analysis using triple quadrupole platforms with offline sample preparation. The method described here utilizes LC-MS(/MS) with an Orbitrap instrument using high resolution accurate mass and online sample preparation. This work will describe a method to screen and quantitate 27 antibiotics at ppb and sub ppb levels in drinking water using online pre-concentration together with high resolution accurate. A standard curve with 27 antibiotics was spiked in neat solution ranging from 100 - 250 ng/mL levels and injected in triplicate. Screening of different water samples (municipal tap water and bottled water) for the targeted 27 antibiotics. The samples was also screened for other unknown pesticides. The data was collected in full MS for generating calibration curves and data dependant all-ion-fragmentation was compared to a current MS/MS library for confirmation for targeted compounds. The limits of detection varied from 100 to 500pg/ml based on the individual compounds with 1 mL injection. For confirmation the exact mass, its isotope pattern and as well as the MS/MS spectrum produced were collected and compared against a fragmentation library which prevented "false positives" in the results. Data mining is one of the main challenges using a high resolution accurate mass system. We will show the data to screen, quantify and confirm in a single run therefore, spending less time on the instrument. Furthermore, the online pre-concentration step allows for low detection limits of these compounds without the time consuming steps of offline solid phase extraction of 1L sample volumes.

P62 Use of D6 Internal Standards for NCI-GC-MS Analysis of Eight Pyrethroids in Environmental Samples

Del A. Koch[1] and Kevin Clark[2]

[1]**ABC Laboratories,** 7200 E. ABC Lane, Columbia, MO 65202, USA; kochd@abclabs.com; [2]Morse Laboratories, LLC, 1525 Fulton Avenue, Sacramento, California 95825, USA; clarkk@morselabs.com

The vulnerability of gas chromatographic methods to signal enhancement or suppression due to matrix co-extractives, and drift over time due to other parameters, is well known. Particularly in cases where the need for low detection limits require a large concentration factor from the sample to the final extract, some levels of matrix co-extractives remain even after the sample cleanup procedures. The nature of complex environmental samples (such as biosolids and wastewaters) often preclude the use of matrix-matched standards as a corrective for matrix effects because of the difficulties in obtaining controls which are analyte-free while at the same time mimicking the unknown sample compositions. Also, the use of matrix-matched standards does not account for instrumental drift with time. The use of deuterium-labeled isotopic analogues of eight targeted pyrethroid insecticides to improve method accuracy and performance when applied to the analysis of environmental samples by negative chemical ionization gas chromatography with mass spectrometric detection (NCI-GC-MS) will be described. In addition, an alternate use of the D6 standards – the determination of method recoveries at low levels that would normally have been precluded due to background pyrethroid levels present in environmental samples being used for control fortifications – will be proposed.

P63 Insecticide Usage Pattern on Brinjal and Resistance in Brinjal Shoot and Fruit Borer (BSFB) and Residue in Brinjal

Dr. Md. Arifur Rahman, Senior Scientific Officer, Entomology Division, Bangladesh Agricultural Research Institute, Gazipur, Bangladesh

Attempt was made to investigate the on-farm situation of insect pests of brinjal with management of BSFB and resistance of BSFB and status of residue levels of 5 insecticides was determined in five brinjal growing areas of Bangladesh.

Farmer's could recognize 4-7 insect and mite pests depending on study area. All confirmed BSFB as the key pest of brinjal. Twenty insecticides with 5 of carbamate 8 of organophosphate and 7 of pyrethroid were acknowledged. Eleven insecticides were found every location with 100% incidence of malataf, diazinon, cypermethrin and fenvalerate. Most popular among 0-5 day interval between insecticide application was 5 day. Farmers apply insecticide 20-150 times per season and maximum farmers preferred 34 times. Zero to 5 day gap between insecticide application and marketing of brinjals, most farmers market at 1-2 day after spray. Among the four methods to control, 100% farmers liked insecticide application over other methods. Among four different bioassay methods, leaf round method showed very consistent results with below 90% and above 70% mortality of 2nd instar larvae of BSFB and the Standard Error was almost always the lowest. Per cent resistance of BSFB of five location varied greatly according to test insecticides and location. Residues of all the 5 insecticides were found up to 10 days. Residue amount in general was above Maximum Residue Limit (MRL) upto 5 days for all insecticides. Fifty one among 100 brinjal samples contained residue of one or two of the test insecticides. Forty were single and 11 were double contaminated. Among the contaminated samples 75%-93.33% samples contained above MRL residue.

P64 Development of a GC-MS/MS Based MRM Method for Quantitation of PCB and PBDE Congeners in Human Plasma

Yanping Lin[1]; Birgit Puschner[1]; Helen (Qingyu) Sun[2]; Gwen Lin[3]; Keyu Zhou[4]; Kefei Wang[2]

[1]Department of Molecular Biosciences, School of Veterinary Medicine, University of California, Davis, California, USA; [2]Bruker Daltonics, Chemical Applied Markets Division, Fremont, California, USA, helen.sun@bruker.com; [3]Life Science Solution, Kuala Lumpur, Malaysia; [4]Bruker Daltonics, Singapore

Polychlorinated biphenyls (PCBs) and Polybrominated diphenyl ethers (PBDEs) are two series of persistent organic environmental pollutants (POPs) origin from industry products. Recent studies have suggested the presence of PCBs and/or PBDEs in the blood or tissues of exposed mothers is correlated with an increasing risk of Autism spectrum disorders in children. Therefore, determination of the quantitation levels of PCBs and PBDEs in human has become part of the comprehensive study for Autism disorder. In the current work, a GC-MS/MS based multiple reaction monitoring (MRM) method for simultaneous quantitation of more than 20 PCBs and PBDEs in human plasma sample has been developed. PCBs and PBDEs were extracted and cleaned-up from plasma with an optimized SPE procedure, followed by GC-MS/MS analysis using a Bruker SCION™ TQ GC triple quadrupole mass spectrometer system. PCBs and PBDEs were separated on a 30 m x 0.25 mm x 0.25 µm GC column using a 27 minute temperature ramping program ranging from 90 to 310 °C. Positive EI ion source was employed to generate ions for MS analysis. The currently developed GC-MS/MS MRM method quantifies each PCB or PBDE congener compound using two MRM transitions. Validation data showed that this method is sensitive with limits of quantitation for most of the PCBs and PBDEs in the range of 0.02-0.05 pg/µL (ppb), and with linearity up to 100 pg/µL. The Bruker SCION™ TQ GCMS system demonstrates excellent signal reproducibility even after hundreds of injections of plasma matrix samples. The finalized MRM method has now been applied to a clinical study of more than two hundred plasma samples.

P65 Determination of 4-Nonylphenol in Biosolids and Biosolids-Applied Soil Using SpeedExtractor and GC/MS/MS

Kang Xia,[1] Huiqin Guo,[1] William H. Ickes[2]

[1]**Virginia Tech**, 185 Ag Quad Lane (241 Smyth Hall), Blacksburg, VT 24061, USA; kxia@vt.edu; [2]Buchi Corporation, 19 Lukens Drive, Suite 400, New Castle, DE 19720 USA

4-nonylphenol (4-NP) is metabolite of nonylphenol polyethoxylates which are anthropogenically-produced substances utilized in many consumer products. Because of its close association with household products, large quantity of 4-NP, which is highly hydrophobic, can be quickly sorbed by the organic rich solid phase during wastewater treatment. Concentrations as high as several thousand mg/kg (ppm) have been detected for 4-NP in biosolids, making biosolids a possible major source for 4-NP in the environment. Land application of biosolids as soil amendment has been a cost-effective biosolids management option for many municipalities. Biosolids production in the United States reached 8.2 million dry ton/year in 2010, 70% of which was land applied. However, there is a strong concern over potential pollution of the environment with various biosolids-associated anthropogenic chemicals such as 4-NP. 4-NP can bind to estrogen receptors, making it a threat to various organisms in the environment. There is a need for fast, accurate, and sensitive detection of 4-NP in environmental samples. In this study, a quantitative method was developed for 4-NP analysis in biosolids and soil samples using a pressurized solvent extractor (Buchi SpeedExtractor E-916) for efficient extraction of 4-NP from the sample matrix followed by detection on GC/MS/MS (Agilent 7890A). Recovery of 105 ±12% and 98±5% were achieved for biosolids and soil samples, respectively. The limit of quantitation (LOQ) was 0.02 ppb for biosolids and 0.01 ppb for soils. The SpeedEXtractor could extract 6 samples simultaneously resulting in high sample throughput with ~ 5 min extraction time/ sample.

P66 Use of Banana Peel for Removing Atrazine and Ametryne from Natural Waters

Sérgio H. Monteiro, Graziela C. R. M. Andrade, Claudineia R. Silva, Taciana F. Gomes and Valdemar L. Tornisielo; Center for Nuclear Energy in Agriculture, **University of Sao Paulo**, Av. Centenário, 303; Piracicaba, SP 13416-903, **BRASIL**; 55 19 34294764; shmonteiro@cena.usp.br

Use of natural materials such as *e.g.* grape bagasses, rice husks, watermelon shelves and bio-organisms for pesticide removal from surface and groundwater, thus solving a serious environmental problem, has been increasing during recent years. In this regard, banana peel presents favorable characteristics, being already used for reducing heavy metal contamination in wastewaters. The aim of this work was to demonstrate the feasibility of using banana peel for removing the pesticide atrazine and ametryne from natural water resources near intensely cultivated areas in order to propose an efficient, fast and low-cost alternative for polluted water remediation. For the pesticide determinations, a 1200 series HPLC in tandem with a 6410 series triple quadrupole MS (both from Agilent Technologies) were used. Due to the good sensitivity of this equipment (LOQs = 0.10 and 0.14µg L^{-1} for atrazine and ametryne) sample preconcentration was not required. The analytical curves were linear up to 20µg L^{-1}, precise results (r.s.d. < 4.5 %) were obtained and recovery data (three addition levels) ranged from 83 to 107%. The solutions with the removed pesticides were stable during three weeks when kept in refrigerator. The best conditions for pesticide removal under laboratory conditions were: sample volume = 50 mL; mass of banana peel = 3.0g, shaking time = 40 min. The pH adjustment was not needed. Analysis of the results revealed a 90% removal efficiency for both pesticides, and real samples were properly remedied demonstrating that banana peel is an efficient stuff for treatment of natural waters polluted with pesticide residues.

P67 Development of QuEChERS/Low-Pressure Fast GC-MS/MS Method for the Analysis of Tris(2-Chloroethyl)Phosphate in Rice

Thanutchaporn Semathong, Alexandre Oglobline and Natchanun Leepipatpiboon; **Chromatography and Separation Research Unit, Department of Chemistry, Faculty of Science, Chulalongkorn University,** Bangkok 10330, Thailand; Natchanun.L@chula.ac.th

Tris(2-chloroethyl)phosphate (TCEP) is a flame retardant compound that drew much international attention due to possible toxicity to humans. It is included in REACH regulation to be banned in EU and is recommended to be eliminated in Norway by 2020. TCEP was found in wildlife and environmental samples in Canada, 0.55 ppb in seagull eggs, in Norway, 0.3 ppb in capelin and in USA, 1,950 ppb in pine needles. We develop a method for the quantification of TCEP in rice using QuEChERS extraction based on EN 15662 protocol with Low-Pressure Fast GC-MS/MS detection. The retention time (t_R) of TCEP was 3.8 min and the GC cycle time was 8 min when analysed as part of the multiresidue screen. The t_R can be reduced to 2 min if only one analyte is required. The 249>187 and 251>189 m/z transitions were used for the quantitation and confirmation respectively. As TCEP produces very weak signal of the molecular ion in EI GCMS, the positive samples could be confirmed by LC-MS/MS using molecular ion $[M+H]^+$ transitions 285>223 and 285>161 m/z. The LOQ of the QuEChERS/Low-Pressure Fast GC-MS/MS method was in order of 0.003-0.01 mg/kg that was set

to comply with uniform default regulatory limit of Japan and can be lowered down to 0.001 mg/kg if extract is concentrated. The recoveries of TCEP were in the range 80%-110% depending on rice variety.

P68 Advances in Automating Solid Phase Extraction for UCMR3: Hormones and 1, 4-Dioxane

Michael Ebitson[1], David Gallagher[2]

[1]**Horizon Technology Inc.**, 45 Northwestern Dr, Salem, NH 03079 USA, mebitson@horizontechinc.com; [2]**Horizon Technology Inc.**, 45 Northwestern Dr, Salem, NH 03079 USA, dgallagher@horizontechinc.com

Currently the U.S. EPA is proposing monitoring for the third Unregulated Contaminant Monitoring Regulation (UCMR3). This action, taken every five years, establishes a program for no more than 30 contaminants in drinking water. Laboratories that have been approved for this program by the EPA by passing their proficiency testing can take place in this program from 2013-2015. The objective of this project is to create two automated SPE extraction procedures that are fast and provide consistent recoveries within their respective recovery acceptance criteriaâ€™s. This research project will be focusing on seven hormones (17-?-estradiol, 17-?-ethynylestradiol (ethinyl estradiol), 16-?-hydroxyestradiol (estriol), equilin, estrone, testosterone and androstene-3,17-dione) based off of method 539 and one compound (1,4-dioxane) from method 522. Both methods have different solid phase extraction and analysis procedures. Method 539 is based off of 47mm spe disks and requires analysis by liquid chromatography electrospray ionization tandem mass spectrometry (LC-ESI-MS/MS) and 522 is based off or cartridges and requires analysis by gas chromatography by mass spectrometry with selected ion monitoring. (GC/MS by SIM). This paper will explain new developments and procedures in automated SPE technology, methodology and chemistry. These new developments will not only provide a solution for processing drinking water samples with two different automated SPE extractors, but in fact provide an increase in throughput, recoveries and consistency over manual SPE procedures.

P69 Mitigation of Off-Site Mobility of Dimethazone and Trifluralin Herbicides

George F. Antonious, **College of Agriculture, Food Science, and Sustainable Systems**, Division of Environmental Studies, Kentucky State University, Frankfort, KY 40601, USA; E-mail: george.antonious@kysu.edu

Nine biobeds (ground cavity filled with a mixture of composted organic matter, topsoil, and a surface grass) were established at Kentucky State University research farm (Franklin County, KY) to study the impact of this practice on reducing surface runoff water contamination by residues of dimethazone and trifluralin herbicides arising from an agricultural field. Biobed (biofilter) systems were installed at the bottom of the slope of specially designed runoff plots to examine herbicides retention and degradation before entering streams and rivers. In addition to biobed systems, three soil management practices: municipal sewage sludge (SS), SS mixed with yard waste compost (SS+YW), and no-mulch rototilled bare soil were used to monitor the impact of soil amendments on herbicide residues in soil following natural rainfall events. Biobeds installed in NM soil reduced dimethazone and trifluralin by 84 and 82%, respectively in runoff water that would have been transported down the land slope of agricultural fields and contaminate natural water resources. Biobeds installed in SS and SS+YW treatments reduced dimethazone by 65 and 46% and trifluralin by 52 and 79%, respectively. Biobed systems could be used to intercept pesticide-contaminated runoff from agricultural fields, creating optimum conditions for sorption and biodegradation such that the amount of pesticides adjacent to water bodies is significantly reduced. This may provide a potential solution to pesticide contamination of surface and groundwater from farmlands.

P70 Comprehensive Analysis of Persistent Organic Pollutants Using High Performance Time-of-Flight Mass Spectrometry

David E. Alonso and Joe Binkley, LECO Corporation, 3000 Lakeview Ave., St. Joseph, MI, USA; david_alonso@leco.com

Environmental samples often contain a multitude of compounds in a wide range of concentrations. Unfortunately, these samples are analyzed for one or two specific classes of compounds. This leads to inaccurate toxicity reports since the samples may contain additional hazardous materials present at higher concentrations than targeted compounds. Mass spectrometers with increased selectivity and sensitivity, as well as, comprehensive capabilities would be ideal for the analysis of these complex samples. In this study, high performance TOFMS with resolving power up to 50,000 allowed for analysis of compounds in dirty matrices, facilitated comprehensive analyses of environmental samples and provided high quality data for library searches. In addition, mass accuracy values better than 1 ppm allowed for robust elemental composition determinations. Targeted and non-targeted analyses were performed on the resulting data files. This allowed for identification of targeted compounds (e.g., dioxins, PCBs), but also polyaromatic, halogenated polyaromatic and pesticide contaminants in sediment and biota samples.

P71 Evaluation of Measuring Pyrethroids in Sediment Using Solid Phase Extraction and GC/MS-NCI and GC/MS/MS-EI

Jona Verreth and Angela Schaner, Montana Department of Agriculture, MSU, McCall Hall, Bozeman, MT 59715, USA; jverreth@mt.gov

Nationwide there is a growing interest in the presence of pyrethroids in the environment particularly sediment. The combination of pyrethroids, which are active at ultralow trace levels and the complexity of a sediment matrix results in an analytical challenge. This method evaluates the efficiency of a Soxtec apparatus with the effectiveness of a double SPE cleanup (Dual layer ENVI-Carb II/PSA 500/500 mg and alumina neutral 1000 mg) and detection options of both EI and NCI source. A summary of the results with its successes and challenges ahead are presented. Recovery studies were performed by fortifying three sediment matrices at LOQ and 10 x LOQ concentrations. The method detection limits ranged from 0.070 to 3.0 µg/kg in dry sediment. Cis$^{13}C^{6}$-permethrin was used as surrogate standard and Trans$^{13}C^{6}$-permethrin as internal standard.

P72 Efficient Pesticides Remediation using OPH enzyme bearing Metal Organic Framework (MOF)

Yongwoo Lee, Ura Shin, and Lawrence Renna, **QinetiQ North America, Inc**., 360 2nd ave., Waltham, MA 02451, USA; yongwoo.lee@qinetiq-na.com

Regenerative, sorption-reinforced catalytic system capable of detecting and decomposing a range of pesticides including methyl parathion (MPT) was developed to provide comprehensive protection against pesticides contamination. Our approach consists of embedding enzymes into the desired substrate to create durable material that detect and fully degrades pesticides, and sequesters both degradation products [Dimethyl Thiophosphate (DMTP) and p-nitrophenol (pNP)] so that the material can be recycled or safely disposed of. Organophosphorous hydrolase (OPH, EC.8.1.3.1) was employed to catalyze the hydrolysis of methyl parathion (MPT). Organophosphorous Hydrolase (OPH) was incorporated into absorbing metal organic framework (MOF) to create the catalytic system. Organophosphate degrading metal organic framework (MOF) was also synthesized and implemented into the catalytic system, separately. Cross-linked poly-β-Cyclodextrin (PCD) having a bifunctional hexyl spacer were prepared as a physical support in 2nd line of defence which is chemically inert in acidic and basic media while preserving accessibility of methyl parathion (MPT) and its degraded product p-nitrophenol (p-NP). It was demonstrated that the OPH enzyme bearing polymeric systems were capable of rapid response to and degradation of methyl parathion (MPT).

P73 Lowering Detection Limits for 1,4-Dioxane in Drinking Water Using Concurrent Solvent Recondensation - Large Volume Splitless Injection in an Unmodified Split/Splitless GC Inlet

Jack Cochran, Chris English, and Chris Rattray, **Restek Corporation**, 110 Benner Circle, Bellefonte, PA, 16823, USA; jack.cochran@restek.com

Global concern over the carcinogenic potential of 1,4-dioxane, along with its identification as a Group 2B compound by the World Health Organization's International Agency for Research on Cancer, has led to increased regulatory interest in this compound. As part of Unregulated Contaminant Monitoring Rule 3 (UCMR3), the U.S. EPA is requiring increased monitoring of 1,4-dioxane in drinking water and has revised the $1x10^{-6}$ cancer risk assessment level down to 0.35 µg/L, which is the basis for the UCMR3 proposed minimum reporting level for 0.07 µg/L.

Large volume injection is advantageous for trace-level contaminant analysis because it increases the amount of target compound introduced to the GC column. Usually, a specialized injector (e.g. a PTV) is required, but it is not appropriate for the very volatile 1,4-dioxane if solvent removal is attempted. Concurrent solvent recondensation – large volume splitless injection (CSR-LVSI) does not have this limitation, since the whole sample goes into the GC column, and the technique can be accomplished in an unmodified splitless GC inlet. CSR-LVSI requires fast autosampler injection with liquid sample band formation into a liner containing glass wool, a retention gap press-fitted to the analytical column, and a starting GC oven temperature below the boiling point of the solvent. We use it to report better GC-MS detection limits for 1,4-dioxane and results for drinking water samples extracted according to EPA Method 522, "Determination of 1,4-Dioxane in Drinking Water by Solid Phase Extraction (SPE) and Gas Chromatography – Mass Spectrometry (GC-MS) with Selected Ion Monitoring (SIM)".

P74 The Holy Grail: Comprehensive Polyaromatic Hydrocarbon Analysis by Serial Combination of HPLC Columns with Different Selectivities and UV and Fluorescence Detection

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

Polyaromatic hydrocarbon analyses are performed regularly throughout the world for both food safety and environmental testing. Many organizations test for PAH residues in food products but the analytes lists vary from organization and country. For example, domestically, the EPA, FDA, AOAC and NOAA all test for PAHs but do not share the same target compound lists. Internationally, the EU and individual countries will test for different compounds. Chromatographic methods are typically tailored to specific target compounds and are not transferable when target compounds change. A further complication is the need to separate interfering compounds occur in real samples, like triphenylene, perylene and benzo[e]pyrene. We developed a chromatographic solution that allows the separation of twenty-nine PAH compounds including interfering compounds. This was accomplished by combining two HPLC columns in series; a PAH specific phase and an aromatic bond selective phase. Separate column selectivities were tested and then combine based on a predicted ratio of the column lengths that would results in no coelutions. Temperature was also determined to play a critical role in the elution order and resolution of the PAHs tested. Time programmed fluorescence detection was optimized to yield the best sensitivity possible, ppb range, with closely eluting compounds.

P75 Characterization of Crude Oils and Tar Balls using Biomarkers and Comprehensive Two-Dimensional Gas Chromatography

Michelle Misselwitz, Jack Cochran, Chris English, and Barry Burger, **Restek Corporation**, 110 Benner Circle, Bellefonte, PA, 16823, USA; michelle.misselwitz@restek.com

The analysis of petroleum biomarkers are commonly used to determine the origin of crude oil samples. These biomarkers are "molecular fossils" which are complex hydrocarbon molecules from formerly living organisms in the source rock. Utilizing biomarkers to fingerprint crude oils is advantageous because they are resistant to weathering or degradation and provide a unique profile for oil to oil correlations. This information can be used by environmental forensic chemists to pinpoint source oil from spills, or in this case, tar balls washed along the shore of the Gulf of Mexico.

Gas chromatography mass spectrometry (GC-MS) is typically used to monitor the ratios of hopane and sterane isomers. This work will focus on characterizing crude oil samples, including from the broken riser pipe collected via an underwater robot during the

Deepwater Horizon oil spill of 2010, by comprehensive two-dimensional gas chromatography time-of-flight mass spectrometry (GC x GC-TOFMS). GC x GC is a two-dimensional separation using two columns of a different selectivity with thermal modulation. Utilizing a retention plane provides a structured chromatogram that aids in fingerprinting complex oil samples. The hopane/sterane profile of these oils will then be compared to extracts of tar balls that washed ashore on the Gulf of Mexico beaches months after the spill occurred.

P76 Determination of PAHs in Fish by QuEChERS Extraction and Dual Layer SPE Cleanup

Xiaoyan Wang and Michael J. Telepchak, UCT, 2731 Bartram Rd., Bristol, PA 19007, USA; xwang@unitedchem.com

In this study a new, sensitive, and reliable method has been developed for the determination of sixteen US EPA priority PAHs (Polycyclic Aromatic Hydrocarbons) in fish. PAHs are extracted into acetonitrile with addition of magnesium sulfate and sodium chloride, the extract is then cleaned up with a novel dual layer SPE cartridge containing primary secondary amine and endcapped C18. GC/MS in SIM mode was used to detect and quantify the sixteen PAHs. Excellent recoveries ranged from 82.6 to 110.9% with relative standard deviations less than 4.1 % were achieved. The dynamic linearity ranges were from 2 to 1000 ng/g with average correlation coefficient of 0.9964. The limit of quantifications were less than 1.57 ng/g for all target PAHs, which are lower than the EU's maximal level of 2 ng/g Benzo[a]pyrene in fish and much lower than the US FDA level of concern of 35 ng/g Benzo[a]pyrene in finfish. This method is sensitive, reliable, and inexpensive, uses GC/MS equipped with a normal splitless injector, offers an attractive alternate for many environmental and food safety laboratories to determine PAHs in fish without investing for more sensitive and expensive analytical instruments.

P77 Estimation of measurement uncertainty for determination of pesticide residues in soil by liquid chromatography-mass spectrometry

Diego. A. Ahumada F.[1], Nicolas Vanoy V.[2], Sandra C. Crespo[3], Danny Rodriguez L.[4], and Adriana Zamudio;[1]

[1]**Laboratorio de Inocuidad Química.** Centro de Bio-Sistemas. Universidad de Bogotá Jorge Tadeo Lozano, Bogota D.C,Colombia; diego.ahumadaf@utadeo.edu.co; [2]GICIC. Facultad de Ingeneria Química.Universidad Nacional del Litoral, Santa Fe, Argentina; [3] Laboratorio de Fisiología y Bioquímica Vegetal. Universidad Nacional de Colombia, Bogotá D.C, Colombia; [4] Unidad de investigación y desarrollo. Agrilab LTDA. Bogota D.C., Colombia.

The correct interpretation of a measurement result requires knowledge about its uncertainty. Very different approaches for the estimation of the uncertainty related to measurement results are found in the literature and in published guidelines. In this work two different approaches for uncertainty estimation are compared on an easy, rapid and low-cost sample preparation approach for the determination of pesticide residues in soils by ultrafast liquid chromatography coupled to mass spectrometry. The first method was based on the guide to the expression of uncertainty in measurement (GUM) approach, which derives the uncertainty of a measurement result by combining the uncertainties related to the uncertainty sources of the measurement process. The second method was an approach previously presented by Haslôva and Stêpán, which combine the botton up and top down methods. The results showed that when is employing GUM approach the uncertainty values were lowest compared with Haslôva method. Using the GUM approach uncertainty of standard preparation (uncertainties of weighing and diluting standards, uncertainties of purity of standards) was shown to represent the main source of combined standard. On the other hand, when the second method was used, the main source of uncertainty extraction process was identified. By contrast, it was found that the source contributes less to global uncertainty was the standards preparation. Finally, the expanded uncertainties for each pesticide using a coverage factor of two were between 13.9% and 25.5%.

P78 Sorption and desorption of ametryn and glyphosate applied in association in soil of sugarcane cultivation: Could a molecule interfere on the behavior of the other in association?

Paulo A. de Toledo Alves,[1] Valdemar L. Tornisielo,[1] Eloana J. Bonfleur,[2] Ana Carolina R. Dias,[1] and Franz Z. Vilca; [2]

[1]**Center for Nuclear Energy in Agriculture CENA/USP**, Avenida Centenário 303, Piracicaba SP - BRASIL; ptoledo@cena.usp.br; **[2]**Escola Superior de Agricultura Luiz de Queiros ESALQ/USP, Avenida Padua Dias S/N, Piracicaba SP – BRASIL

The environmental behavior of pesticides depends directly on its usage and form of application in crops. Sorption and desorption characteristics are very important to understand the persistence and the mobility of the molecule in the environment. The aim of this study was to determine the sorption and desorption of ametryn, glyphosate and ametryn+glyphosate (tank mix simulation) in soil of sugarcane cultivation, using as guideline the OECD 106 for testing chemicals. The test was performed using ^{14}C radiolabeled techniques, in 8 concentrations (1/2, 1, 2, 4 and 8 times the field doses). The soil characteristics were determined by the ESALQ soil department: pH (in $CaCl_2$) 5.93; Total Sand ($g.Kg^{-1}$) 582; Clay ($g.Kg^{-1}$) 302; and Organic Matter ($g.dm^{-3}$) 17. The experiment was set in Teflon tubes of 50 mL volume (in triplicates), with 0.2 g of soil and work solution (in $CaCl_2$ 0,01 mol L^{-1}) in 5:1 proportion (solution:soil). The equilibrium time was set in 24h during horizontal agitation (200 rpm 20 ºC). The centrifugation was performed at 3000 rpm (4 ºC) for 15 minutes. The results were read in LSC and the Freundlich model was applied to sorption and desorption data. The statistic paired T test was used to evaluate differences between treatments and doses. Individually, glyphosate reached high K_{oc} and low K_{ds}, contrasting with the ametryn results (low K_{oc} and low K_{ds}). Ametryn did not influenced glyphosate sorption or desorption. On the other hand glyphosate increased the ametryn sorption 5x, also decreasing its desorption 4x.

P79 Reduction of Mancozeb Residues on Tomatoes by Dipping Solutions

Mehmet F. Cengiz and Muharrem Certel, Akdeniz University, Faculty of Engineering, Food Engineering Department, Antalya/TURKEY; fcengiz@akdeniz.edu.tr

Mancozeb is a broad spectrum contact fungicide with a protective action which belongs to the dithiocarbamates family of chemicals. The official statistics on the use of plant protection products in the European Union indicate that, mancozeb is in the second row of the top ten fungicide active ingredients list. This active ingredient is a source of ethylenethiourea which is also a suspected carcinogen. Therefore, there is an increasing need to develop methods to reduce mancozeb residue in widely consumed food product to acceptable levels. Today, the use of dipping solutions has shown potential as an effective postharvest treatment in the reducing pesticides residue. In this research, the effects of chlorine and hydrogen peroxide on the reduction of mancozeb residues on/in tomato samples were studied. Two different concentrations (10 and 100 ppm) of each chemical were tested for the predetermined reaction time (0, 5, 15, and 20 min). After the reaction time, the tomatoes were removed and immediately analysed for the determination of remaining mancozeb residues by using a GC/MS system. The results showed that the waiting for 20 min in 100 ppm Chlorine dipping solutions was the most effective in removing mancozeb residues, with an average of 70,8% of the residues being eliminated from the samples, followed by hydrogen peroxide solutions with an average of 64,7% at the same time and concentration. Water was least effective application for the reduction of mancozeb residue. These experiments indicated the potential for the removal of pesticide residues on tomato samples.

P80 Determination Of Organoclhorine Pesticides In Sea Turtle Liver Using A Multiresidue Method and GC-µecd

Angélica María Sánchez-Sarmiento [(1)]; Franz Zirena Vilca [(2)]; Silmara Rossi [(3)]; José Henrique Becker[(4)]; Juliana Marigo[(1) (5)]; Valdemar Luis Tornisielo [(2)] & Eliana Reiko Matushima [(1)]

[(1)] Department of Pathology, College of Veterinary Medicine and Zoothechny, University of São Paulo, São Paulo, BRASIL - Prof. Dr. Orlando Marques de Paiva Avenue 87, CEP 05508-270; ang.san.sar@gmail.com; [(2)] Center of Nuclear Energy in Agriculture (CENA/USP), University of São Paulo, Piracicaba, BRASIL - Centenário Avenue 303, CEP 13400-970, PO Box 96. [(3)] "Luiz de Queiroz" College of Agriculture - Center of Nuclear Energy in Agriculture (ESALQ/CENA/USP). [(4)] TAMAR-ICMBio Project. [(5)] BioPesca Project, Santos, São Paulo, BRASIL

Sea turtles are included in the IUCN Red List of Threatened Species, this threats include catch by fisheries, habitat degradation in the feeding and nesting areas and diseases. Some impacts are related to the negative effects of pollutants, like the organochlorines compounds. A new method for the analysis of organochlorine pesticides (α- BHC, β - BHC, heptachlor, dicofol, OP´ DDD, α - endosulfan, OP´DDE, PP´DDE, β - endosulfan, PP´DDD, endosulfan sulfate, myrex and dieldrin) residues in sea turtle liver samples was developed. It involved extraction of 1 g of liver with acetonitrile saturated n-hexane (18%), shaken 1 min; centrifuged at 3000 g for 15 min at 5°C twice; the organic phase was freezed for 15 min at -15°C and transfered to 50 mL tube containing 1200 mg of $MgSO_4$ and 400 mg of PSA, mixed 1 min, centrifuged at 4800 g for 15 min at 15°C collected 4 mL, evaporated with nitrogen and resuspended in 1 mL of n-hexane, transfered to a column of silica gel for a second clean up, collected and evaporated with nitrogen, resuspended in 1 mL of toluene and injected into the GC–µECD system. The extraction method was adequate and the validation process is currently being worked. The monitoring of these compounds can provide information about the possible relation of these residues with adverse effects in sea turtle health such as the fibropapillomatosis in *Chelonia mydas*.

P81 Monitoring Of Organoclhorine Pesticides In Green Sea Turtle Fat Using A Multiresidue Approach Method and GC-µecd

Angélica María Sánchez-Sarmiento [(1)]; Franz Zirena Vilca [(2)]; Silmara Rossi [(3)]; José Henrique Becker[(4)]; Juliana Marigo[(1) (5)]; Valdemar Luis Tornisielo [(2)] & Eliana Reiko Matushima [(1)]

(1) **Department of Pathology, College of Veterinary Medicine and Zoothechny**, University of São Paulo, São Paulo, BRASIL - Prof. Dr. Orlando Marques de Paiva Avenue 87, CEP 05508-270; ang.san.sar@gmail.com (2) Center of Nuclear Energy in Agriculture (CENA/USP), University of São Paulo, Piracicaba, BRASIL - Centenário Avenue 303, CEP 13400-970, PO Box 96. (3) "Luiz de Queiroz" College of Agriculture - Center of Nuclear Energy in Agriculture (ESALQ/CENA/USP). (4) TAMAR-ICMBio Project. (5) BioPesca Project, Santos, São Paulo, BRASIL

Sea turtles are included in the IUCN Red List of Threatened Species, this threats include catch by fisheries, habitat degradation in the feeding and nesting areas and diseases. Some impacts are related to the negative effects of pollutants, like the organochlorines compounds. A new method for the analysis of organochlorine pesticides (α- BHC, β - BHC, heptachlor, dicofol, OP´ DDD, α - endosulfan, OP´DDE, PP´DDE, β - endosulfan, PP´DDD, endosulfan sulfate, myrex and dieldrin) residues in green sea turtle fat samples from São Paulo coast was developed. It involved extraction of 1 g of fat with acetonitrile saturated n-hexane (18%), shaken 1 min; centrifuged at 3000 g for 15 min at 5°C twice; the organic phase was freezed for 15 min at -15°C and transfered to 50 mL tube containing 1200 mg of $MgSO_4$ and 400 mg of PSA, mixed 1 min, centrifuged at 4800 g for 15 min at 15°C collected 4 mL, evaporated with nitrogen and resuspended in 1 mL of n-hexane, transfered to a column of silica gel for a second clean up, collected and evaporated with nitrogen, resuspended in 1 mL of toluene and injected into the GC-µECD system. The extraction method was adequate and the validation process is currently being worked. The monitoring of these compounds can provide information about the possible relation of these residues with adverse effects in sea turtle health such as the fibropapillomatosis in *Chelonia mydas.*

P82 Polybrominated Diphenyl Ethers (PBDEs) in Surface Sediments of Hillsborough Bay, FL

Candice Simmons[1], Foday Jaward [2]

[1]**College of Marine Science**, University of South Florida, 830 1st Street South, St. Petersburg, FL 33701, USA; ccsimmo2@mail.usf.edu; [2] Department of Environmental and Occupational Health, College of Public Health, University of South Florida, 13201 Bruce B. Downs Blvd., MDC 56, Tampa, FL 33612, USA

Polybrominated diphenyl ethers (PBDEs) are a widely used class of flame retardants that are important sources for contamination in the marine environment. Sediments serve as a sink for PBDEs due to their large sorption capacity, and concentrations of PBDEs in

sediments are related to coastal population density. Hillsborough Bay, a northeastern region of Tampa Bay, FL, is surrounded by a large urban area, supports extensive industrial activity and a major shipping port. This study examines the presence and distribution of PBDEs in Hillsborough Bay to determine the extent of pollution, identify sources, and possible management strategies. A total of 50 sediment surface samples were collected from Hillsborough Bay and two of its major tributaries to assess the presence and distribution of 8 routinely detected PBDE congeners. Samples were soxhlet extracted, cleaned up on a multilayer silica gel column and extracts were analyzed by gas chromatography/ mass spectrometry (GC/ MS). In this study we report the first attempt to present data and distributions of PBDEs in terms of sample location, grain size and organic matter content in this region.

P83 Fate of Imidacloprid and its Metabolites in Honey Bee Hives

Alaa Kamel[1], Galen Dively[2], David Hawthorn[2] and Jeff Pettis[3]

[1]Analytical Chemistry Laboratory, Biological and Economic Analysis Division, Office of Pesticides Program, US EPA, 701 Mapes Road, Fort George G. Meade, Maryland 20755, USA, email: kamel.alaa@epa.gov; [2]Department of Entomology, University of Maryland, College Park, Maryland; [3]Bee Research Laboratory, Agricultural Research Service, USDA, Beltsville, Maryland

The fate of imidacloprid and its metabolites in honey bee hives treated for six weeks with 100 ppb of imidacloprid in a pollen diet or 20 ppb of imidacloprid in a sugar syrup diet has been determined. These amounts simulate residue levels that were found in a previous study to be present in the pollen and nectar of pumpkin flowers treated with product label application rates of imidacloprid. Samples of worker honey bees, bee larvae, royal jelly, honey and bee bread were collected from each hive at 2 and 6 weeks of treatment and 6 weeks post treatment. Samples analysis was carried out using LC-MS/MS and showed that honey samples contained the highest levels of imidacloprid, followed by bee bread, royal jelly and adult bee samples in hives treated with 100 ppb imidacloprid in pollen diet. In hives treated with 20 ppb imidacloprid in sugar syrup diet, traces of imidacloprid between LOD and LOQ (0.2-0.6 ppb) were detected only in honey samples. No imidacloprid metabolites were found in any of the samples.

P84 Benefits and Limits of an LC-MS/MS Based Online Sample Clean-up Method for High Throughput Analysis of Pesticide Residues in Different Food Matrices with Regards to the Latest Carbendazim Contamination Issue in Orange Juice

Laszlo Hollosi,[1] Klaus Mittendorf,[1] Hamide S. Senyuva[2]

[1] **Food Safety Response Center**, Thermo Fisher Scientific GmbH, Im Steingrund , Dreieich, D-63303, Germany ; laszlo.hollosi@thermofisher.com; [2] FoodLife International Ltd, Ankara Turkey

Pesticide residue analysis is still a challenging area of food testing and benefits from new analytical method development. The need for faster, less laborious, but on the other hand still precise and robust multi-residue methods with high recovery and sample throughput still remains high priority. It is therefore attractive to consider options for automation of as many steps of a multi-residue method as possible. For this purpose online sample clean-up methods are potential options. This presentation describes advantages and limits of an on-line sample clean-up method for the simultaneous determination of 48 pesticides (including carbendazim) in grapes, baby food, orange and wheat flour matrices. Target pesticides were selected to represent a wide variety of chemical structures in polarity range -2.0<log P< 6.9, while selected matrices represented the typical consistency forms of samples and the latest concern of orange. Turbulent flow chromatography was applied for on-line sample clean-up and analyte pre-concentration directly coupled to liquid chromatography-tandem mass spectrometry. The aim was to reduce total analysis time, eliminate manual laboratory work, provide clean extracts and reproducible results. Single laboratory validation of the method was conducted establishing limits of detection to be between 0.3-5.0 ng/g, within-day precision values between 4 and 18%, while between-day precisions between 5-22%. Method recovery ranged from 67 to 124 %, and method accuracy was demonstrated by analysis of external quality control samples (Fapas). The presentation aims to give more detailed information on benefits and limits of this 13 min total run time method.

P85 A New Sample Preparation Method for Malachite Green and other Food Dyes in Salmon by LC-MS-MS

Elena Gairloch[1] and Mats Leeman[2]

[1]**Biotage**, 10430 Harris Oaks Blvd, Suite C, Charlotte, NC 28269, USA, Elena.Gairloch@Biotage; **[2]MIP Technologies AB**, A subsidiary of Biotage AB, Box 737. SE-220 07 Lund, Sweden, Mats.Leeman@biotage.com

Malachite green (MG) and crystal (gentian) violet (CV) are triphenylmethane dyes used in aquaculture as fungicides and ectoparasiticides. They are rapidly metabolised to the leucomalachite green (LMG) and leucocrystal (gentian) violet (LCV) metabolites that persist in fish tissue. MG and CV are banned in many countries due to their mutagenicity and carcinogenicity, but may still be used illegally.

Monitoring methodologies must be able to detect the compounds at 1 ng/g (US) or 2 ng/g (EU). For the leuco forms, which have poor absorbance in the UV and VIS range, this becomes difficult on LC-UV/VIS instruments. Instead LC-MS-MS is the analytical method of choice due to its sensitivity, selectivity and ability to detect compounds which lack good UV absorbance.

Traditionally, sample preparation of the fish extracts have been done by liquid-liquid extractions using relatively large volumes of organic solvents such as dichloromethane, followed by sample clean-up on one or more sorbents. While the methods often have good performance, they may be both time consuming and use larger volumes of environmentally and hazardous solvents.

The presented method focuses on ease of use when compared to other methods, and describes a QuEChERS based approach incorporating the use of aluminium oxide in enhanced dispersive SPE to reduce the levels of fat in the final extract. Additional

cleanup is provided by polymeric EVOLUTE SPE columns. The resulting extract demonstrated low ion suppression and analyte recoveries of 60-105%, with low rsds. The method is also applicable to Brilliant green

P86 Monitoring of Pesticide Residue in Agricultural Products using GC/MS/MS

Nam Suk Kang, Seong Cheol Kim, Won Jo Choe, Jong Hoon Ahn, Kwang Soo Yoo, Jung Eun Lee, Dong Yun Jung, Rack Seon Seong, Ki Sung Kwon; Busan Regional Korea Food and Drug Administration, 65, 356-Gil, Shinseon-Ro, Nam-gu, Busan, Korea; kangns97@korea.kr

This monitoring study was investigated to collect data on pesticide residue levels in agricultural products from South Korea. A total number of 52 samples of 15 agricultural products collected from 9 regions. 183 kinds of pesticides were analysed using multi-residue analytical method in these sample. In perilla leaf of the samples was detected diniconazole. The residue level was 0.578 mg/kg, exceeded maximum residue limits (MRLs). For this pesticide, the average recoveries were found to be 119.8% (RSD = 0.017%, n = 3). Procymidone was detected in 3 strawberries (0.068 ~ 0.920 mg/kg) and one watermelon (0.154 mg/kg), but within safe levels, i.e. not exceeded MRLs for pesticides in foods by the Korea Food Code. In 47 samples no residues were detected.

P87 High Throughput Quantitative Analysis of Pesticides by Gas Chromatography with Triple Quadrupole Mass Spectrometry

Ryan Picou, John Reuther, Eurofins Analytical Labs, 2315 N. Causeway Blvd.; Metairie, LA 70001, USA; ryanpicou@eurofinsus.com

As a contract testing lab, high throughput and high quality analysis is critical for success. The data presented here demonstrate a method validated in our laboratory that meets these needs. This method has been optimized for speed of analysis. A challenge with fast chromatographic separations is determining an adequate data scan rate to represent narrow peaks for accurate quantitation. In this GC-MS/MS acquisition method, eight pesticides are analyzed within a 10 minute gas chromatographic separation. Two MRM transitions are assessed for each pesticide. This presentation will describe how the scan rate was adjusted to ensure adequate data points for quantitation, which is applicable to any chromatographic analysis. Specific to this presentation, each pesticide's dynamic range is presented. Also, the precision of sample preparative method as well as the data acquisition robustness are indicated by assessing fortified corn samples at three concentrations over three days.

P88 Internship Program - Unique Opportunity for Future Scientists

Yelena Karaseva, U.S. FDA, Northeast Regional Laboratory, 158-15 Liberty Ave; Jamaica, NY 11433; yelena.karaseva@fda.hhs.gov

The US FDA Northeast Pesticide Laboratory is a leader in the Internship program between FDA and York College-CUNY. Promoting fundamental science interests with emphasis in the food and health care areas is central to the missions of both FDA and York College. Both organizations are committed in improving the quality of and access to scientific research training and their proximity offers students a unique opportunity to engage in joint collaborative activities. The York College/FDA student internship is an ideal way for students to find instruction and mentorship in an area of academic and personal interest while also preparing for a future career through work experience and networking opportunities. Thirty interns for the past five years had the unique opportunity to gain hands-on training in a wide variety of methods, tests, and procedures to determine contamination of foods in pesticide analysis. They developed practical skills such as homogeneous sample preparation, liquid/liquid partitioning, solid phase extraction, quality assurance and quality control laboratory practices and documentation of analytical results. They enhanced their theoretical knowledge of analytical chemistry, gas chromatography (GC), liquid chromatography (LC) and basic mass spectrometry (MS) together with applied interpretation of different GC/MS chromatograms in trace pesticide residue analysis. Currently, intern students participate in basic research projects for regulatory method development. Specifically, modifications of AOAC QuEChERS method have been evaluated in various food matrices for the enhancement of diazinon and endosulfan in pesticides recoveries and their results will be presented in this forum.

P89 An automated, chronic, pesticide bio-assay, based upon reductions in *Vibrio fischeri* luminescence, can be used to screen for the presence of Dibrom toxicity in natural waters, with an EC_{50} of ~0.4ppm.

Albaro Perez, Patricia Barreto, Joshua Leibowitz, Vladimir Orlov, and Jose Barreto, **Florida Gulf Coast University (FGCU)** Green Technology Research Group and Department of Chemistry, 10501 FGCU Blvd South Fort Myers FL 33965 USA; *Corresponding author: jbarreto@fgcu.edu

Dibrom is an organophosphate pesticide that kills by blocking synaptic transmission in the nervous systems of higher animals. Dibrom does not persist in the environment, notably, it hydrolyzes in water with a $t_{1/2}$= ~15 hours. Nevertheless, annual applications in the US exceeds one million pounds, and a simple, inexpensive screening assay would be useful, in order to monitor any deleterious acute environmental effects in natural waters, and allay public fears about toxicity. *V. fischeri* are luminescent marine bacteria that demonstrate reduced light output upon killing or injury; these luminescent bacteria are intoxicated by Dibrom, even though they lack a synaptic nerve transmission mechanism. Previously, we developed an automated, high through-put, acute *V. fischeri* bio-assay with a 5 min exposure time for toxins. Dibrom toxicity can be measured using our automated acute assay, but we hypothesized that a chronic toxicity assay would be more sensitive to Dibrom (using ~22 hrs of toxin exposure). We grew *V. fischeri* in plastic 24 well plates and measured light output directly from each well with a TECAN well plate reader. For our chronic assay 'twin wells' were created, wherein a zero toxin control accompanied each 'twin' intoxicated sample; high light output ratios of 0.80-1.2 from twin wells indicated 'no toxicity'; low fractional ratios (<0.80), indicated high toxicity. We now report an EC50 value of ~14.1 ppm for acute studies, with an EC50 value of ~0.40ppm for our chronic studies. The new chronic assay is therefore ~35X more sensitive than the acute assay for the detection of Dibrom toxicity in water.

P90 Automated Extraction and Analysis of Mycotoxins from Food Products Using DPX-LC/MS/MS

Oscar Cabrices[1], Edward Pfannkoch[1], John R. Stuff[1], Fred F. Foster[1], B.J. Bench[2] and William E. Brewer[3]

[1]GERSTEL Inc., Linthicum, MD, USA; [2]Tyson Foods Inc., Springdale, AR, USA; [3]Department of Chemistry and Biochemistry, University of South Carolina, Columbia, SC, USA; ogcabrices@gerstelus.com

One of the major food safety challenges is the abundance of mycotoxins, including but not limited to *Aspergillus flavus* and *Aspergillus parasiticus* molds, which are among the fungi that produce toxic secondary metabolites known as aflatoxins. Several mycotoxins are known to be human carcinogens or to cause other human disease. Mycotoxin levels in food and animal feed are regulated in most countries and there is great interest in a fast, sensitive, and selective analysis method. Determining mycotoxin concentrations at trace levels in the presence of large amounts of sample matrix is a challenging task. The accuracy and precision of the analytical results depends largely on the extraction and cleanup methods used to isolate the mycotoxins from the complex food and animal feed matrices.

In this work, the automated extraction of mycotoxin residues from contaminated corn samples using disposable pipette extraction (DPX) is described. DPX is a solid-phase extraction (SPE) technique that is based on loosely contained sorbent inside a pipette tip fitted with a screen. This device provides faster extraction because only minimal conditioning steps are needed. The automated DPX extraction and analysis method for a panel of 11 mycotoxins (aflatoxins, fusarium toxins and fuminosins) provided extraction efficiencies greater than 70% for all mycotoxins screened in corn samples with RSDs less than 15%. In addition; good linearity was achieved (R2 values of 0.98 or greater) with limits of quantitation lower than confirmation cutoff concentrations for most analytes. The ability to automate the dispersive SPE clean-up of corn sample extracts and to couple the extraction directly to LC/MS/MS analytical methods, results in improved laboratory productivity by streamlining the complete analytical process.

P91 Monitoring of Hexaconazole Residues in Agricultural Products of Korea

In-Kyung Kim, Myung-Sang Yoo, Myoung-Jin Go, Eun-Heui Park, Soo-Kyung Suh; **Gwangju Regional Korea Food & Drug Administration**, 315 Cheomdangwagiro, Buk-gu, Gwangju, Korea; kik82@korea.kr

Hexaconaozle is a fungicide used for the control of a various kind of fungi in agricultural products. While this hexaconazole has used in recent years in korea, the monitoring data of hexaconazole residues in agricultural product was not reported yet. The purpose of this study was to monitor the level of hexaconazole residues in 15 different agricultural products(6 vegetables, 8 fruits and rice) which were most widely consumed in Korea. Hexaconazole were determined by gas chromatography coupled with electron capture detector. The standard curve showed a linear detection range between 0.01 through 5 ug/mL (R^2=0.9998). The limit of detection (LOD) and limit of quantification (LOQ) were 0.005 and 0.02 mg/kg, respectively. The average recoveries and the relative standard deviations(RSDs) ranged from 70.88 to 78.87% and from 0.60 to 7.31 in rice, green & red pepper(fresh), mandarin, potato, peanut and soybean. No Hexaconazole residue was detected in 156 samples. These results will be used as a fundamental database for risk assessment of pesticides in Korea.

P92 A Rapid Method for the Determination of Multi-Mycotoxins in Wines and Beer by LC-MS/MS using a Stable Isotope Dilution Assay

Fadwa Al-Taher[1], Katie Banaszewski[1], Jerry Zweigenbaum[2], and Jack Cappozzo[1]

[1]**Institute for Food Safety and Health**, 6502 S. Archer Rd, Bedford Pk, IL 60501, USA; altaher@iit.edu
[2]**Agilent Technologies, Inc.,** 2850 Centerville Rd, Wilmington, DE 19898, USA

A multi-mycotoxin dilute and shoot method for the liquid chromatography-tandem mass spectrometric (LC-MS/MS) determination of mycotoxins in wines and beer has been developed and validated. Separation was accomplished using ultra-high performance liquid chromatography (UHPLC) by a linear water-methanol gradient with < 10 min analysis time. Mycotoxins were detected by dynamic multiple reaction monitoring in positive electrospray ionization mode. Due to matrix effects, [^{13}C]- uniformly labeled mycotoxins for 11 of the 13 compounds tested were added to the sample extracts prior to LC-MS/MS analysis. With external calibration, the obtained apparent recoveries were 18 - 148% for white wines, 15 - 118% for red wines, and 20 - 125% for beer when spiked at three levels. The [^{13}C]- labeled internal standards compensated for matrix effects effectively with overall recoveries 67-103% for white wines, 70-120% for red wines and 61-125% for beers, with greater recoveries for FB1 and FB2, at three spiking levels. The relative standard deviation was <20% for all analytes in the wines and beer. The trueness of the method was confirmed for ochratoxin A (OTA) by testing a reference material. This method was applied to a USDA-funded nationwide survey of domestic and imported wines and beer for the determination of OTA and extended to include all the other validated mycotoxins.

P93 Determination of Antibiotic Residues in Milk and Dairy Products

Tom Dobbs, Jessica Netzer, Jeff Wiseman, Jennifer Salmons, **J2 Scientific,** 1901 Pennsylvania Drive, Suite C; Columbia MO 65202 USA; tdobbs@j2scientific.com

The presence of antibiotics in food stuff is of great concern since they are a potential health risk due to promotion of wide spread bacterial resistance to antibiotics used in humans, possible allergic reactions, and carcinogenicity. In many cases the fermentation process in the production of cultured dairy products can be adversely affected by traces of antibiotics in raw milk, resulting in considerable economic loss. Reduction of human exposure to antibiotics has led many regulatory agencies to develop maximum residue limits for each of these substances to protect human health.

There is a need for a fully automated method for the detection of beta-lactam antibiotics including penicillins and cephalosporins in milk that is reliable, meets the MRL requirements, and is inexpensive. A simple method has been explored for simultaneous determination of penicillins and cephalasporins which consists of the simultaneous solid phase extraction using C18 cartridges, and a strong polymeric mixed mode anion exchanger. Quantitation is accomplished using HPLC with UV-Vis detection. Method performance parameters such as recoveries, detection limits, and reproducibility are discussed.

P94 Selective and Sensitive Analysis of Organophosphorus Pesticides in Baby Food Using an Inexpensive GC Detector

Scott Hazard and Gary Engelhart, **OI Analytical**, 151 Graham Road, College Station, TX 77842-9010, USA

Organophosphorus (OP) pesticides are cholinesterase-inhibiting insecticides which have been widely used in agriculture due to heir broad spectrum activity and effectiveness. Studies have demonstrated that consumption of food items containing OP pesticide residues is the predominant pathway of exposure in children. The National Research Council has reported that the vulnerability of infants and children is characterized by immature physical development and high consumption of fresh fruits and vegetables. Many analytical methods for OP pesticide residues are based on the QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) approach followed by GC/MS or GC/MS-MS detection and quantitation. The presence of pigments, fats, proteins, and other matrix interferences can obscure MS peak identification, and the cost of a triple quadrupole MS can be prohibitively expensive and thus fails the criterion for a "Cheap" analysis. This poster describes the use of a Pulsed Flame Photometric Detector (PFPD) in phosphorus-selective mode for sensitive, selective detection and quantitation of OP pesticide residues in baby food. Comparative PFPD and MS chromatograms for a variety of baby food extracts are presented.

P95 Analysis of Antioxidants in foods and Dietary Supplements Using HPLC with Post-Column Derivatization

Wendy Rasmussen, Research Chemist, wendyr@pickeringlabs.com and Maria Ofitserova, Ph. D. Senior Research Chemist, **Pickering Laboratories, Inc.** Mountain View, California 94043, USA. (650)964-6700, maria_o@pickeringlabs.com

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

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

P96 High Throughput LC-MS-MS Analysis of Carbendazim in Orange Juice

Jeffrey H. Dahl and William Hedgepeth, **Shimadzu Scientific Instruments**, 7100 Riverwood Drive, Columbia, MD 21046, USA; (410) 381-1227 ext. 1843; jhdahl@shimadzu.com

Carbendazim is a fungicide used on cereal and fruit crops. Although its use is no longer permitted in the US, there are a number of countries that still legally use Carbendazim. Recently, orange juice imported from BRASIL has tested positive for Carbendazim. A number of commercial orange juices were tested by our newly developed method and found to contain Carbendazim in low ppb levels. Orange juice samples were diluted 40 to 50x with water and centrifuged before analysis. Isocratic and gradient reversed phase high speed methods were developed for the analysis of Carbendazim on a 2.0 x 50 mm 1.6 micron column coupled to a triple quadrupole mass spectrometer. A high throughput injector that features a seven second injection time was used to help speed the sample throughput. Three commercial brands of orange juice tested positive for Carbendazim at low ppb levels. An organic brand of orange juice did not contain any detectable levels of Carbendazim and was used to prepare the matrix matched calibration curves. Good linearity was obtained from low ppb levels up to a 1 ppm concentration. A blank injected after the 1 ppm standard showed no carryover. The use of UHPLC with a high speed injector allowed an analysis to be completed within a one minute timeframe.

P97 Increasing speed of UHPLC-MS analysis using single-stage Orbitrap mass spectrometer

Maciej Bromirski and Olaf Scheibner, **Thermo Fisher Scientific**, Hanna-Kunath-Str. 11**,** Bremen 28199, Germany; maciej.bromirski@thermofisher.com

Productivity of a LC-MS system is measured in samples per day. To accomplish high productivity, modern UHPLC-MS methods increasingly deal with very short gradients, leading to chromatographic signals with peak widths below 5 seconds at the base. It is still a challenge for HRMS systems to provide a sufficient number of scans (≥10) across the chromatographic peak in full scan mode without compromising sensitivity and selectivity.

Horse feed samples were spiked with commonly occurring pesticides and veterinary drugs and injected onto a UHPLC system (Thermo Scientific Accela™ system) coupled to a single-stage Orbitrap mass spectrometer (Thermo Scientific Exactive™Plus). For chromatographic separation a Thermo Scientific Hypersil Gold aQ column (100x2.1mm, 1.9µm) was used. The mass spectrometer was run with a resolution setting of 70,000 (FWHM @ m/z 200).

Quantitative and qualitative analysis was run on pesticides and vetenary drugs in horse feed matrix, using a calibration range from 1 ppt to 500 ppb. Dilution series were measured to generate calibration curves and differently spiked samples were measured. The

data rate with respect to the peak width was observed. Running the mass spectrometer at a resolution setting of 70,000 resulted in a data rate of 3.7 scans per second, so that the limit of 10 scans across the peak was reached even for peaks being as narrow as 3 seconds at the base. Combination of the quantitative data processing with a general screening approach using Thermo Scientific ExactFinder™ software yielded the detection of additional components present in the unknown samples. Results were confirmed by isotopic pattern matching of the proposed sum formulas and library searching.

P98 Detection and Quantitation of Brominated and Chlorinated Hydrocarbons by DART with Linear Ion Trap and Triple Quadrupole Technology

Cory Lytle[2], Michal Hauer[2], Mary Blackburn[1], Scott J. Harrison[3], Maria C. Prieto Conaway[1]

[1]Thermo Fisher Scientific, 355 RiverOaks Parkway, San Jose, CA 95134; [2]**Thermo Fisher Scientific**, 1400 North Pointe Parkway, West Palm Beach, FL; [3]AgResearch, Palmerston North, New Zealand

Brominated hydrocarbons also known as brominated flame retardants (BFRs) have been used in various industries for decades. Recently several classes of BFRs have been detected in the biosphere.Chlorinated hydrocarbons have also been used for many years, primarily as pesticides (OCs), the most infamous of these being DDT. While most OCs have been banned in the United States, their use still occurs in devoloping countries. The continued use of BFRs and OCs as well as their persistance in the environment and potential deleterious activity therein makes the detection and monitoring of these compounds an important topic. We propose Direct Analysis in Real Time (DART) as a simple, rapid, easy to use technique for detection and quantitation of both BFRs and OCs. Negative ion full scan mass spectral data was acquired on the LTQ linear ion trap. Sequential losses of halogen were observed when MSn data was acquired on the linear ion trap for the BFRs and OCs. Using the preliminary data acquired on the linear ion trap precursors and transitions were chosen for quantitation using the Quantum Access triplequad. Calibration curves were created from stock solutions starting at 1ppm and ranging down to 100ppt. LODs, LOQs, and linearity was determined based on the analysis of 5 replicates at each level. In conclusion, each sample requires approximately 6 seconds for analysis, sample preparation is minimal and DART optimization is very easy.

P99 Evaluating the Effectiveness of Incorporating Ceramic Homogenizers in the QuEChERS Extraction Method

Brian Kinsella, Xiaoyan Wang, and Thomas August, **UCT Inc.**, 2731 Bartram Road; Bristol, PA 19007, USA; bkinsella@unitedchem.com

The QuEChERS sample preparation method was introduced in 2003 by Anastasiades *et al.* for the analysis of pesticide residues in food. It involves extracting and partitioning residues from an aqueous sample into acetonitrile with the addition of salts to induce phase separation. This is followed by dispersive solid phase extraction (dSPE) for the removal of matrix co-extractives. Since its inception, there has been a dramatic increase in the use of QuEChERS, including in pesticide, veterinary drug and environmental analysis. Numerous modifications have been introduced into the method to improve clean-up and recovery (e.g. buffered extraction and different dSPE sorbents). Recently, ceramic homogenizers have been introduced to enhance recovery and decrease variability of the QuEChERS method. The aim of this study was to evaluate QuEChERS extraction with and without ceramic homogenizers. Three different matrices and two classes of residues were evaluated. The extraction of 16 polycyclic aromatic hydrocarbons from fish resulted in lower recovery (overall 6% decrease) and more variability (2.2 vs. 7.2% RSD) when using ceramic homogenizers. The extraction of pesticides from cereal and strawberries yielded similar recovery and reproducibility. The results of this study illustrate that ceramic homogenizers do not have a significant effect on the recovery or variability of residue concentrations when used in QuEChERS extraction. While in some cases the use of ceramic homogenizers in difficult matrices may provide a better degree of homogenization, for the matrices tested in this study there was no significant differences in the recovery or variability.

P100 A Field Study on the Removal and Destruction of Polychlorinated Biphenyls from Vertical Painted Surfaces

Simone E. Novaes-Card,[1] Hester Groenevelt,[2] Stefanie Secker,[1] Christian A. Clausen,[1] and Cherie L. Yestrebsky;[1]

[1]**University of Central Florida**, 4000 Central Florida Blvd; Orlando, FL 32816, USA; snovaescard@knights.ucf.edu; [2]Geosyntec Consultants, Inc.; Guelph, Canada

Polychlorinated biphenyls (PCBs) are a class of recalcitrant halogenated organic compounds that were once employed as additives in a wide variety of applications. Their manufacture was halted by the US Environmental Protection Agency in 1978, and current regulations state that PCB concentrations above 50 mg/kg require removal or remediation. Researchers at the University of Central Florida have developed a two-step treatment system for PCBs on vertical painted surfaces. A sorbent paste (non-metal treatment system, NMTS) containing solvents, paint softeners, and thickeners is used to draw the PCBs out of the paint. This paste is then removed and returned to the laboratory, where it is combined with zero-valent magnesium, ethanol, and acetic acid to produce an activated metal treatment system (AMTS) and reductively dechlorinate the PCBs. The AMTS can also be produced on-site for use in a process where PCBs are sorbed from the paint and degraded in the same step. Both treatment options were tested in the field at a site near Seattle, WA. A painted concrete wall and a painted metal door were treated with both NMTS and AMTS, and samples of both paint and paste were taken at 2 and 5 days. Treatment with NMTS reduced PCB concentrations in the paint from the wall and door by approximately 94% and 90%, respectively, over 2 days. Within one day of off-site activation with magnesium and acidified ethanol, the concentration of PCBs in these samples was not quantifiable. Treatment with AMTS produced on-site reduced concentrations by 86.2% and 62.4%, respectively.

P101 Development of *In situ* Degradation Technologies for Chlordane Residues

Tamra Legron-Rodriguez, Ariel Gerszuny, Cherie Yestrebsky, **University of Central Florida**, 4000 Central Florida Blvd; Orlando, FL 32816, USA; tamralegron@knights.ucf.edu

Chlordane is a persistent organic pollutant (POP) which was introduced as an insecticide in the US in 1948. Thirty years later the US Environmental Protection Agency (EPA) banned the use of chlordane for food crops, but its use for termite control continued until 1988 at which time all uses of chlordane were cancelled. Due to its persistence, soils contaminated with chlordane residues remain problematic and *in situ* remediation of this widespread contamination would be advantageous. This study describes the degradation of chlordane using an activated metal treatment system (AMTS), a paste containing zero-valent magnesium, ethanol, acetic acid, and appropriate fillers, developed at the UCF Environmental Chemistry Laboratory, which has previously proven successful for the *in situ* remediation of polychlorinated biphenyls (PCBs). This study found that the activated metal treatment system reductively dechlorinated a 2.0 mg/kg chlordane sample by approximately 57% after one day of treatment, 84% after five days of treatment, and below the detection limit after eight days of treatment. This is promising considering the EPA has set safe levels of exposure at 5×10^{-4} mg/kg per day (ingested) and 5×10^{-5} mg/m^3 per day (inhalation) to avoid adverse health effects. The results of this study demonstrate that this treatment system rapidly and effectively remediates chlordane providing a viable *in situ* degradation method. A complete degradation study including kinetic data will be presented.

P102 Self-Carmelization of Sucralose: Identification of over 100 Degradation Products by UHPLC and LC/QTOF-MS with Ultrahigh Resolution time-of-flight mass spectrometry and Accurate Mass

E. Michael Thurman[1], Imma Ferrer[1] Jerry Zweigenbaum[2]

[1]**University of Colorado,** Department of Environmental Engineering, 1100 Engineering Drive, 428 UCB, Engineering Center, University of Colorado, Boulder, CO 80309 USA, mthurman@ono.com; [2]Agilent Technologies, Inc. Wilmington, DE, USA

Sucralose, a chlorinated sucrose, is an important sugar substitute that is used world-wide in food, drinks, vitamins, and many other food uses. We discovered that sucralose can undergo self-carmelization (self charring to a carbon-like substance) in a sealed container when stored at room temperature for extended periods of time, i.e. more than one year. Apparently, the chlorine atom on the glucose ring is labile and forms hydrochloric acid upon long term storage. The hydrochloric acid is volatile and spreads throughout the solid material causing a blackening of the white sugar and a browning, as well. We extracted the blackened residue and found over one hundred different chlorinated structures, based on the accurate mass of the degradation products and the known starting structure. The most common structure was a three membered ring with the addition of a glucose unit to the sucralose structure. Many isomeric forms were found as the glucose ring is added to different hydroxyl groups on the sucralose structure. We will discuss the process of self-carmelization, the identification of degradation products by UHPLC and LC/Q-TOF-MS, and the possible pathways of formation of these degradation products. Finally, the possibility of this process occurring in food will be discussed along with toxicological implications of self-carmelization of sucralose.

P103 Automated QuEChERS Tips for GC/MS Analysis of Fruit and Vegetables

William E. Brewer, Pakritsadang Kaewsuya, and Stephen L. Morgan, **Dept. of Chemistry and Biochemistry, University of South Carolina**, Columbia, SC 29208, USA; brewerw@mailbox.sc.edu

QuEChERS (Quick, Easy, Cheap, Effective, Rugged, Safe) approaches to analyzing pesticides in fruit and vegetables have become common place. Most approaches utilize a cleanup method prior to chromatographic analysis to further remove fatty acids and unwanted sample matrix components. In this presentation, the cleanup method is performed automated using pipette tips packed with QuEChERS sorbent (75 mg $MgSO_4$, 25 mg PSA, 12.5 mg GCB) and fitted with a filtration screen and transport adaptor. The automated QuEChERS cleanup method was performed using a GERSTEL MPS-2 dual rail sample preparation system equipped with the DPX (disposable pipette extraction) option, and the GC/MS analysis was performed using a GERSTEL 6890 GC with 5975 MSD. The dual rail system utilizes one syringe to perform the cleanup of the original acetonitrile extract (from the QuEChERS approach) and the other syringe to inject into the GC/MS system. In this study, 220 pesticides were analyzed in 5 different sample matices, carrots, tomatoes, celery, broccoli and green beans. Recoveries ranged from 70 to 177% with less than 12% RSDs for most pesticides. The automated cleanup took less than 3 minutes to perform per sample, which easily permits “just in time” sample preparation and even prep ahead features to improve throughput.

Speed up your evaporations

Biotage TurboVap Family – the number one cited evaporator in EPA methods!

TurboVap® II



› Fast evaporation with a single piece of glassware
› Endpoint detection allows unattended evaporation down to 1.0 mL or 0.5 mL
› Up to 6 samples with individual control

TurboVap® LV



› Evaporate up to 50 samples at a time
› Microprocessor-control provides monitoring of timed operations and water bath temperature for fast and efficient evaporation
› Compact and can be placed in a hood or on a bench-top

TurboVap® 500



› Closed cell evaporation system
› Automated sample concentration with solvent recovery
› 2 individually controlled positions

TurboVap® 96



› Compatible with 96-well standard or deep well plates
› 2 positions with individual gas regulation
› Patented vortex evaporation for highest efficiency

Come visit us at
FPRW 2012, booth #16

Biotage | IST

www.biotage.com/sampleprep

ELEVATE CONFIDENCE IN FOOD SAFETY

Confidence means knowing your lab is delivering accurate, consistent, and timely results. We listen carefully to customers' needs, both present and future, engaging in lab-bench to lab-bench scientific collaborations to attack many of the food supply chain's biggest challenges.

Agilent's comprehensive product and service solutions address the discovery and measurement of both chemical and biological contaminant analysis in current and emerging applications across the food spectrum. We're confident that Agilent is uniquely equipped to support food testing– now and in the future.

Access the new Food Safety Applications Compendium at:
www.agilent.com/chem/food

The Measure of Confidence



Food Safety Summit:
Scan with your smart phone to register now.

© Agilent Technologies, Inc. 2012

Agilent Technologies