48th Florida Pesticide Residue Workshop



FPRW 2011

TradeWinds Island Grand Resort
St. Pete Beach, Florida
July 17-20, 2011

"Bringing Scientists Together to Develop and Validate Better Methodologies"

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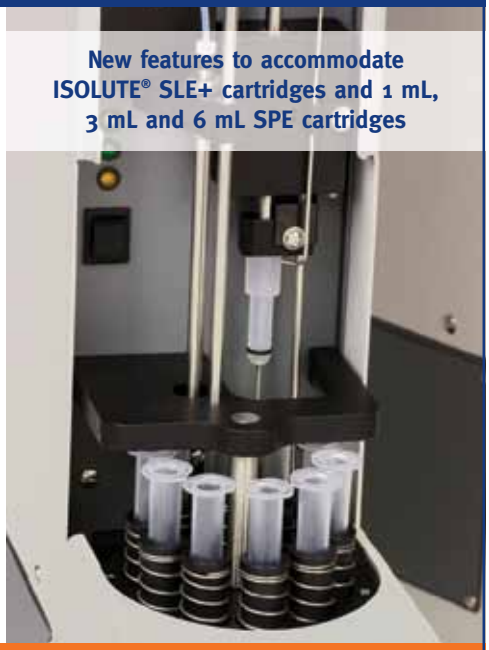
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2011 FLORIDA PESTICIDE RESIDUE WORKSHOP

July 17, 2011

Dear Attendees and Exhibitors,

Welcome to the 48th Florida Pesticide Residue Workshop, the "original" pesticide residue workshop that spawned several others around the world. The California PRW, the European PRW, and the Latin American PRW were all inspired by the FPRW. The first FPRW was organized by George Fong with lots of logistical support from his wife, Wilma. George continued to organize this meeting every year with help from his colleagues at the Florida Department of Agriculture and Consumer Services and others in the food safety community. Even after his retirement in 2000, George continued as the logistical guru and organizer-in-chief who made everything run smoothly. Several years ago, the Florida Department of Agriculture and Consumer Service decided it could no longer sponsor the FPRW. With George's leadership, a non-profit corporation, FLAG Works, Inc. was created with a volunteer Board of Directors. George continued his dedicated service to the FPRW as a part-time employee of that corporation. He has served as the financial officer, liaison to the TradeWinds Hotel, organizing committee member, meeting historian, chief photographer, and overall meeting coordinator. George has been the glue that has held the Florida Pesticide Residue Workshop together for nearly five decades. After 48 years of service to the food safety community, George and Wilma Fong are retiring from their positions of leadership. We want to thank George and Wilma for their inspiration, dedication, hard work and friendship over all these years. Teri Besse, our very capable meeting planner for the last few years, will take over most of George's responsibilities beginning October 2011.

We have added two events to this year's workshop that we hope will be popular. First, we are offering a course on LC/MS/MS techniques taught on Sunday by Dr. Robert Voyksner. Judging by the number of people who have already signed up for this course (in mid June), it looks as though attendees appreciate the chance to learn from one of the leading innovators in LC/MS. The second event is our reception at the new museum dedicated to the work of Salvador Dali. This should be a great opportunity to mingle with your colleagues, meet new people, network with others in the food safety world and see the museum's renowned collection. We really want to thank the vendors who have helped subsidize this event – you'll see their names prominently displayed throughout the meeting.

Once again, we have an outstanding program that fills three full days. Steven Bradbury, Director of the USEPA Office of Pesticide Programs, will be the first in our stellar cast of speakers. There are sessions on effects of the Gulf oil spill, pharmaceuticals and vet drug residues, global harmonization, international monitoring programs and, of course, advances in analytical methods for all kinds of chemical residue analyses.

This year, we had a record number of oral and poster submissions and this posed a dilemma for the program committee. Unfortunately, there was not enough space in the oral program to accommodate all of the submissions and some people were asked to present their work in poster format. With more than 100 poster submissions, we had to display them in two sessions to accommodate them all. Posters are a great way to present your work and to allow for in-depth discussions with the authors. So, please visit the posters and try to speak with the authors of those that interest you.

Our workshop is primarily organized by volunteers who participate on the Organizing and Program Committees. We will have an organizing committee meeting on Tuesday from 5:30 to 6:30 pm in the main meeting room. Please join us for this meeting and get involved! Everyone is welcome to attend and to vote on our slate of officers for the coming years.

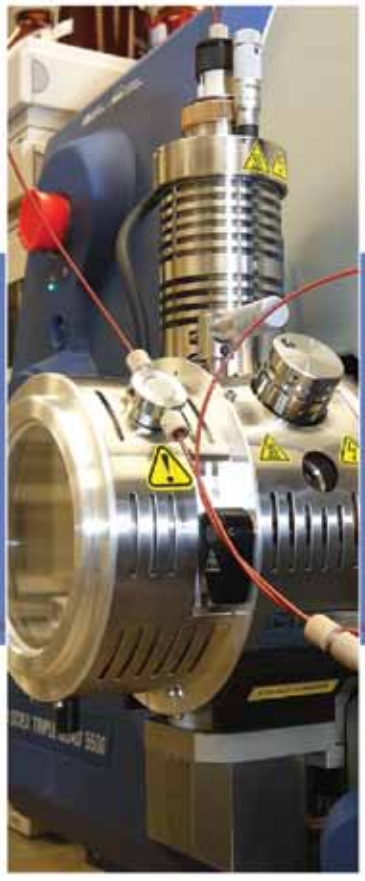
Vendor workshops have always been popular events at the FPRW, both for attendees and the vendors. This year we had so many vendor requests that we decided to offer another slot on Sunday evening. Be sure to attend these because it's often where you hear about the latest instrumentation and techniques for residue analysis.

For many years, the Florida Pesticide Residue Workshop has focused on various chemical residues – not just on pesticides. The analysis of melamine, mycotoxins, PAHs, synthetic hormones, antibiotics and other pharmaceutical residues are hot topics among residue chemists and you will see posters and presentations on all of these issues this week. In recent years, the FPRW has been attracting more and more attendees and presenters from abroad. Their presence enriches our program and broadens our networking opportunities. Recognizing this, the FLAG Works Board of Directors approved a proposal to change the name of the Florida Pesticide Residue Workshop to the North American Chemical Residue Workshop (NACRW). No name can express the complete character of this meeting or its rich history, but we believe that NACRW better reflects its geographical and topical breadth. Furthermore, there is a need for a single meeting at which food safety experts from throughout the region can share new techniques and find answers to current chemical residue problems. We will begin using both names in correspondence and on our website following this meeting. The transition period during which we refer to both names will take two years. The changeover to NACRW is to be fully effective in 2013. This doesn't mean that the meeting has to move from Florida or from the very popular TradeWinds Resort, but it could. In any case, we have signed a contract with the TradeWinds through 2015 so we'll be back here for several more years at least.

Finally, we want to thank all of the vendors for their participation. We owe a special debt of gratitude to those vendors who chose to become meeting sponsors. This extra financial contribution has made it possible to hold the reception at the Dali Museum and has subsidized the food and drink at our breaks. You'll see their names prominently displayed at the meeting. We hope you'll make an extra effort to visit their booths and thank them for their support.

George and Wilma, we will miss you both. Good luck in your second retirement.

Phil Wylie, Lynda Podhorniak, Alaa Kamel, Donna Kilpatrick, Joe Romano, Jack Cochran, Steve Lehotay, and Sherri Turnipseed (2011 Organizing and Program Committee Officers)



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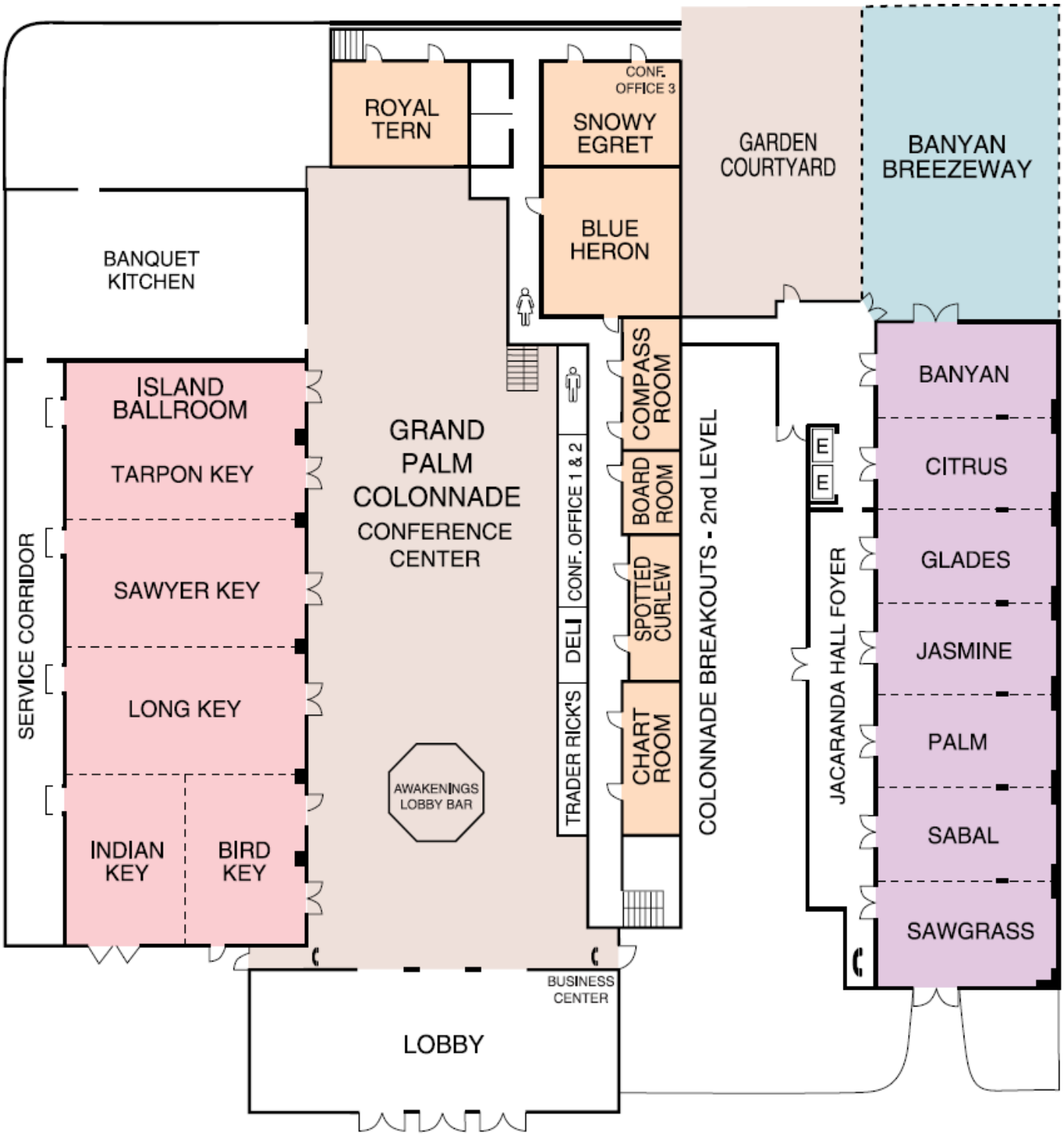


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Technical Sessions: Long Key, Bird Key and Indian Key Ballrooms
Exhibits, Posters, Reception: The Pavilion
Vendor Seminars: Banyan/Citrus Rooms and Horizon Rooms
Sunday Short Course: Snowy Egret

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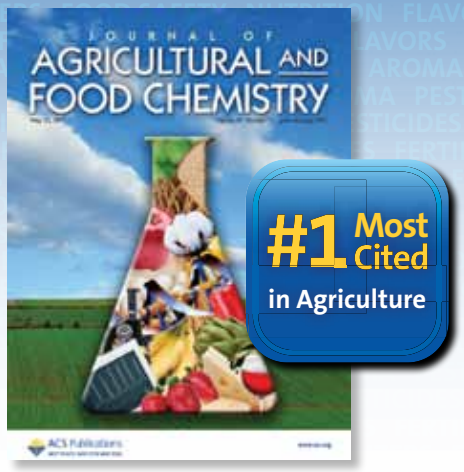
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2011 - 48th Florida Pesticide Residue Workshop

MEETING AT A GLANCE

Sunday, July 17, 2011

8:30 am – 4:30 pm	**Voyksner - LC-MS/MS Short Course**	**Snowy Egret**
5:00 – 8:00 pm	Registration	Grand Palm Colonnade
4:30 – 5:30 pm	Volunteers Orientation	Grand Palm Colonnade
5:30 -10:00 pm	Exhibitor Setup	Pavilion
6:00 – 6:30 pm	Moderator Training	Long/Bird/Indian Key Ballrooms
6:00 pm →	Technical Session Setup	Long/Bird/Indian Key Ballrooms
6:00 pm →	Poster Board Set Up	Pavilion
6:30 – 7:30 pm	**ThermoScientific Vendor Seminar**	**Banyan & Citrus Rooms**
8:00 – 9:00 pm	**Restek Evening Seminar**	**Banyan & Citrus Rooms**

Monday, July 18, 2011

All Day	Registration	Grand Palm Colonnade
7:15 – 8:15 am	Early Morning Coffee	Pavilion & Grand Palm Colonnade
7:15 – 8:15 am	**Waters Breakfast Seminar**	**Horizon Rooms**
8:30 – 11:00 am	Opening Oral Session	Long/Bird/Indian Key Ballrooms
11:00 – noon	Exhibition Opening	Pavilion
11:00 – noon	Poster Session A	Pavilion
12:15 – 1:15 pm	**Bruker Lunch Seminar**	**Horizon Rooms**
1:30 – 3:05 pm	Gulf Oil Spill Session	Long/Bird/Indian Key Ballrooms
3:10 – 3:55 pm	BREAK (Exhibition & Posters)	Pavilion
3:55 – 5:05 pm	Pharmaceuticals & Veterinary Drug Residues	Long/Bird/Indian Key Ballrooms
5:30 pm	Depart for Salvador Dali Museum	TradeWinds Hotel Entrance
9:30 pm	Return from Dali Museum to Hotel	Museum where Buses parked

Tuesday, July 19, 2011

All Day	Registration	Grand Palm Colonnade
All Day	Exhibition & Posters	Pavilion
7:15 – 8:15 am	Early Morning Coffee	Pavilion & Grand Palm Colonnade
7:15 – 8:15 am	**LECO Breakfast Seminar**	**Horizon Rooms**
8:30 – 10:30 am	Global Residue Harmonization	Long/Bird/Indian Key Ballrooms
10:30 – noon	Exhibition & Posters	Pavilion
11:00 – noon	Poster Session B	Pavilion
12:15 – 1:15 pm	**UCT, Inc. Lunch Seminar**	**Horizon Rooms**
1:30 – 3:05 pm	International Monitoring Programs	Long/Bird/Indian Key Ballrooms
3:10 – 4:00 pm	BREAK (Exhibition & Posters)	Pavilion
4:00 – 5:30 pm	Forum on Mass Spectrometry	Long/Bird/Indian Key Ballrooms
5:30 - 6:30 pm	Organizing Committee Meeting	Long/Bird/Indian Key Ballrooms
7:00 - 8:00 pm	Reception by TradeWinds	Pavilion
8:00 pm →	Beach Volleyball Game	On the Beach

Wednesday, July 20, 2011

Until noon	Registration	Grand Palm Colonnade
Until noon	Exhibition & Posters	Pavilion
7:15 – 8:15 am	Early Morning Coffee	Pavilion & Grand Palm Colonnade
7:15 – 8:15 am	**AB Sciex Breakfast Seminar**	**Horizon Rooms**
8:30 – 9:40 am	Advances in Analytical Methods for Chemical Contaminants	Long/Bird/Indian Key Ballrooms
9:45 – 11:00 am	Exhibition & Posters	Pavilion
11:00- 12:10	Advances in Analytical Methods for Chemical Contaminants (cont'd)	Long/Bird/Indian Key Ballrooms
12:20 – 1:20 pm	**Agilent Lunch Seminar**	**Horizon Meeting Rooms**
1:30 – 2:40 pm	Advances in Analytical Methods for Chemical Contaminants (cont'd)	Long/Bird/Indian Key Ballrooms
2:45 – 3:00 pm	BREAK	Grand Palm Colonnade
3:00 – 4:35 pm	Technical Session continues	Long/Bird/Indian Key Ballrooms
4:40 – 5:00 pm	Poster Awards and Closing	Long/Bird/Indian Key Ballrooms

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GENERAL INFORMATION

Registration

Check in once at the registration desk at your earliest opportunity
Sunday - 5:00 – 8:00 pm
Monday - 7:30 am – 5:00 pm
Tuesday - 7:30 am – 5:00 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 numbered, P-1 thru P-57
Session B posters are numbered, P-58 thru P-111

Poster Sessions (in The Pavilion)

Session A - Hang Posters Sunday evening after 6:00 pm or before 8:30 am on Monday
Take down posters by 10:30 am on Tuesday

Session B - Hang Posters Tuesday 10:30 am to 11:00 am
Take down posters by noon on Wednesday

Posters may be viewed any time Exhibition is open

Poster Session A authors must be at their posters from 11:00 am – noon & 3:10 -3:55 pm on Monday
Poster Session B authors must be at their posters from 11:00 am-noon & 3:10-4:00 pm on Tuesday & 9:45 -11:00 am on Wednesday

Poster Prize

Author(s) of Best Poster in each session 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

Exhibition

Monday – 11:00 am - 5:00 pm
Tuesday - 7:30 am - 5:00 pm
Tuesday evening reception with cash bar 6:30 – 7:30 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 be morning and afternoon refreshment breaks each day. All Mon. and Tue. breaks, and the one on Wed. morning, will be served in the Exhibition Tent. On Wed. afternoon, the break will be served in the Grand Palm Colonnade.

Announcements

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

Job Placement Bulletin Board

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

Door Prizes

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

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

You are encouraged to contribute original research and/or review articles to the *Journal of Agricultural and Food Chemistry* for a special section related to FPRW in 2011. Please inform Lynda Podhorniak or Alaa Kamel in advance if you intend to submit an article**.** The manuscripts must be submitted electronically online on the journal's web site by Sept. 1, 2011. Manuscripts will undergo the normal peer-review process to determine if it is acceptable for publication.

Copies of Presentations

Oral Presentations: Following the meeting, if time and resources permit, oral presentations will be posted on our 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 W. George Fong at Gandwfong@comcast.net

Meeting Website

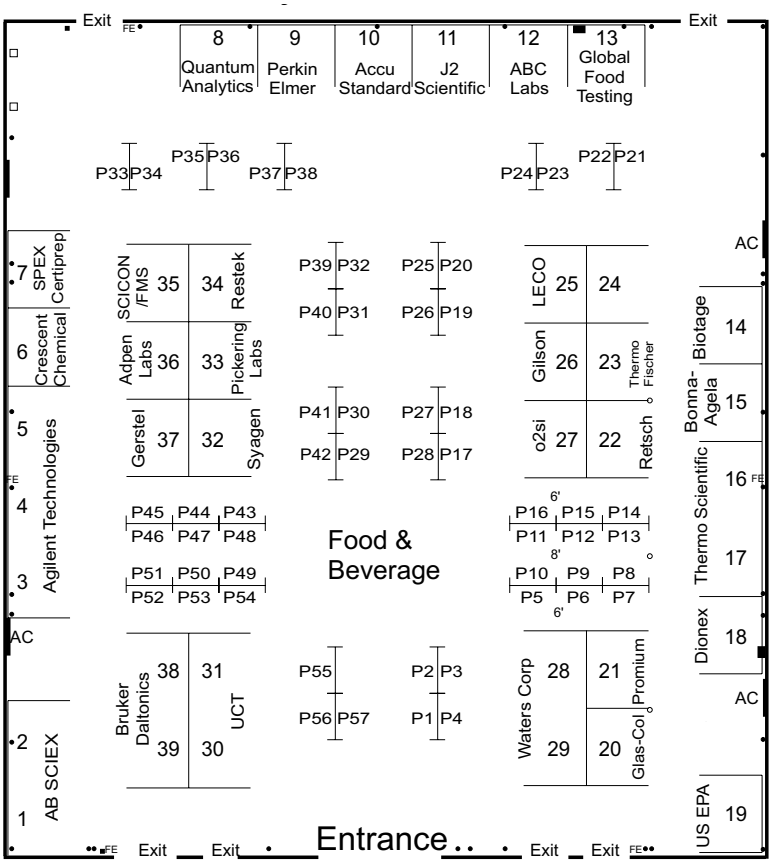
www.flworkshop.com - the website includes information on current and future FPRW meetings, as well as archives going back to 2005.

FUTURE MEETING DATES

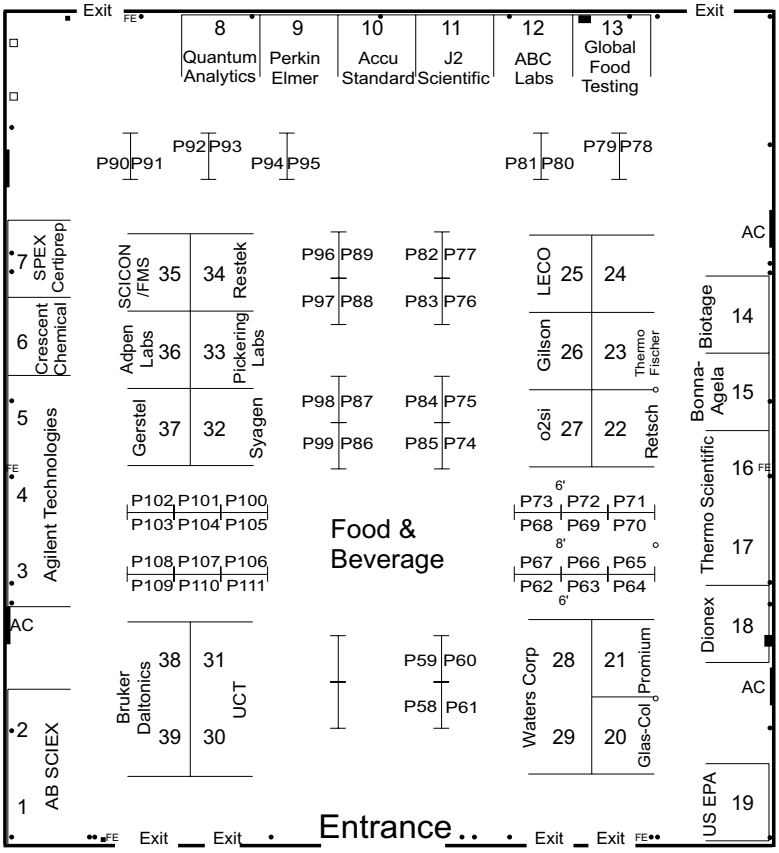
2012 July 15 - 18
2013 July 21 - 24
2014 July 20 - 23
2015 July 19 - 22

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Exhibits and Poster Session A Floor Plan



Exhibits and Poster Session B Floor Plan



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SHORT COURSE
Sunday, July 17, 2011 8:30 am to 4:30 pm
Location: Snowy Egret

Understanding and Optimization of LC/MS/MS to Develop Successful Methods for the Identification and Quantitation in Complex Matrices

Instructor: Dr. Robert Voyksner

This course is designed for the chromatographer / mass spectrometrist who wants to be successful in developing methods, method optimization and solving problems using LC/MS/MS. The course covers the atmospheric pressure ionization (API) techniques of electrospray, pneumatically assisted electrospray and atmospheric pressure chemical ionization (APCI) and atmospheric pressure photo ionization (APPI) using single quadrupole, triple quadrupole, time-of-flight and ion trap mass analyzers. Discussions of sample preparation and chromatography will target method development and optimization for the analysis of "real-world" samples by LC/MS/MS. The course highlights the following topics with respect to optimizing a method to achieve the best sensitivity, specificity and sample throughput: 1) optimization of ionization in API techniques, 2) understanding and minimizing matrix suppression, 3) relative merits of various LC column lengths, particle sizes and column diameters for LC/MS/MS analysis, 4) introduction into the interpretation of MS/MS spectra, 5) important issues in LC/MS/MS quantitation, and 6) optimization of quantitative analysis. Applications of LC/MS/MS to pesticide, herbicides and other hazardous containments in environmental matrices and food products will serve as examples throughout the course to emphasize the topics covered.

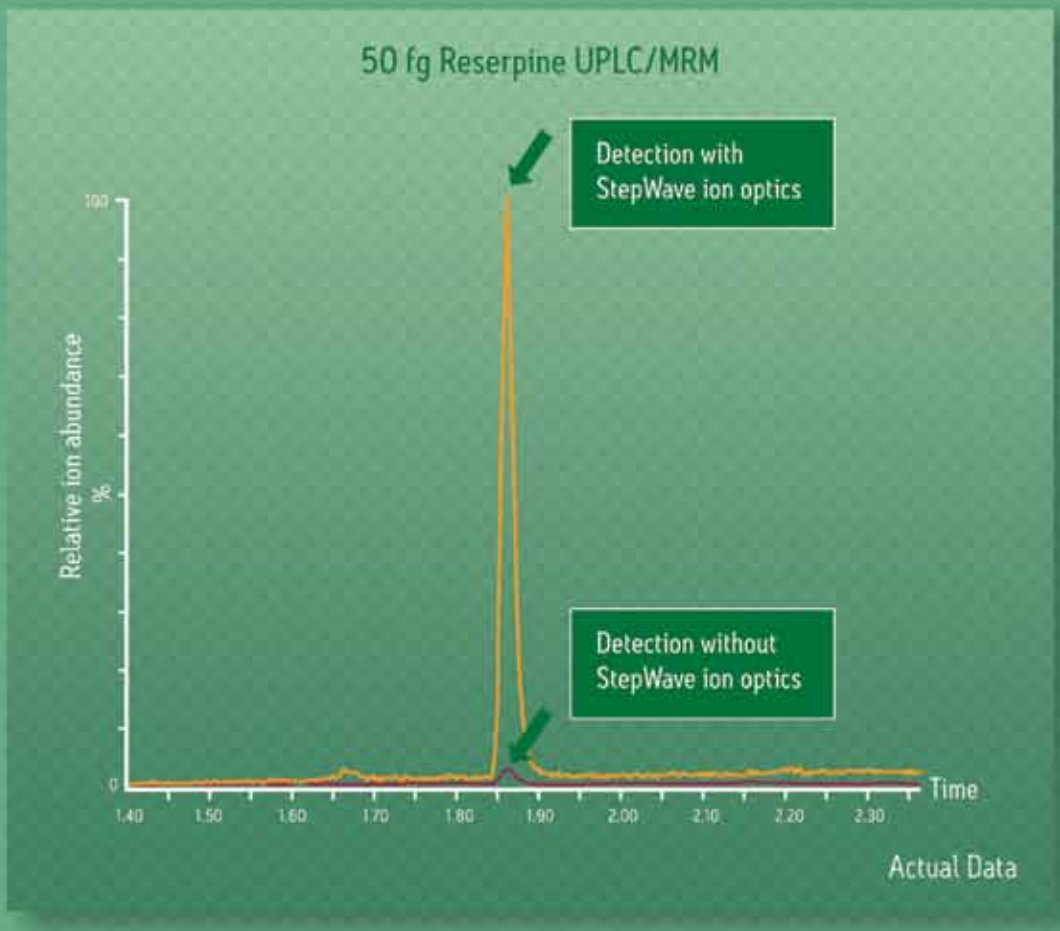
About the Instructor

Dr. Robert D. Voyksner received his B.S. in Chemistry at Canisius College in 1978 and his Ph.D. at the University of North Carolina at Chapel Hill in 1982. He was employed at Research Triangle Institute (RTI) from 1983-2001 as the director of the mass spectrometry facility and has been responsible for developing extraction, separation and mass spectrometric methods for biologically and environmentally significant compounds. His work earned him the Presidents Award, the highest award within RTI. In 2001 he co-founded LCMS Limited in Raleigh, NC and has been the President of the company to date. Under his direction LCMS Limited is working on technological advancements in LC/MS, offering services to pharmaceutical and agrochemical industry for solving unique problems by LC/MS/MS and offering training in LC/MS/MS and MS/MS interpretation and on LC/MS/MS instrumentation. Dr Voyksner is also an Adjunct professor at the North Carolina a School of Veterinary Medicine and at The University of North Carolina School of Pharmacy.

Dr. Voyksner's research in mass spectrometry has resulted in over 230 publications and presentations, primarily in the area of HPLC/MS. He has served on the Board of Directors for The American Society For Mass Spectrometry (ASMS), is on the organization committee for The Montreux LC/MS Symposium and was the organizer for the 1995, 1999, 2003 and 2007 Montreux LC/MS Symposia. Dr. Voyksner has taught over 100 courses on LC/MS, CE/MS and CID interpretation during the past 10 years for ASMS, pharmaceutical companies; ISSX, PBA, HPCE and HPLC focused meetings.

[INTRODUCING XEVO TQ-S]

[You're going to need a bigger graph.]





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VENDOR SEMINARS

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Vendor Seminars: Please sign up at the meeting registration desk

V-1. Sunday Evening, July 17, 2011 6:30 p.m. to 7:30 p.m. Thermo Scientific
Location: Banyan & Citrus Rooms

Non-Target screening with High Resolution Bench Top Orbitrap (Exactive)/SIEVE

James Chang, Thermo Fisher Scientific

Using a novel Bench Top Orbitrap mass spectrometer coupled to U-HPLC, capable of providing quick screen on different food matrices by using high mass accuracy at resolution up to 100,000, which can prevent matrix interference and co-eluting isobaric compounds. In addition to the target compound analysis, a ToxID software can screen unlimited amount of contaminants by providing molecular formula for Know Unknown investigation, a Sieve software was using for totally unknown screen with great success which can provide a tool for detecting the illegal food adulteration such as Melamine in the past.

V-2. Sunday Evening, July 17, 2011 8:00 p.m. to 9:00 p.m. Restek
Location: Banyan & Citrus Rooms

High Quality Analysis of Pesticides in Marijuana for Food and Medicine using QuEChERS, Cartridge SPE Cleanup, GCxGC-TOFMS, and LC-MS/MS.

Jack Cochran, Julie Kowalski, Sharon Lupo, Michelle Misselwitz, Amanda Rigdon, Restek Corporation, 110 Benner Circle, Bellefonte, PA; Frank Dorman, The Pennsylvania State University, University Park, PA

Over 15 states and the District of Columbia have enacted laws that legalize the use of medical marijuana. Potential therapeutic benefits of marijuana have been cited for pain relief, control of nausea and vomiting, stimulating appetite, and muscle relaxation. Unlike other prescribed medicines that are regulated by the US Food and Drug Administration, medical marijuana is still considered a Schedule 1 drug and is therefore illegal on the federal level. Patients have no assurances on the safety of the medicine due to potential harmful levels of pesticide residues.

The Quick-Easy-Cheap-Effective-Rugged-Safe (QuEChERS) extraction approach was applied to marijuana. The complexity of the sample required cartridge solid phase extraction cleanup and comprehensive two-dimensional gas chromatography-time-of-flight mass spectrometry (GC x GC-TOFMS) for pesticide residue analysis. In addition, the sample extracts were diluted and analyzed by liquid chromatography-tandem mass spectrometry (LC-MS/MS) for comparison, and for determination of pesticides such as bifenazate and abamectin that cannot be done with GC.

V-3. Monday Breakfast, July, 18, 2011 7:15 a.m. to 8:15 a.m. Waters Corporation
Location: Horizon Rooms

Use of the Xevo TQ-S as applied to the routine and not so routine analysis of animal tissues for residues of veterinary medicines

Dr. Simon Hird, Head of LC-MS/MS Team, Food and Environment Research Agency (FERA), Sand Hutton, York, North Yorkshire, YO41 1LZ, UK; +44 1904 462567, Simon.hird@fera.gsi.gov.uk

Even with approved use, it is expected that residues will usually turn up in food supplies. Conditions of use are set to ensure that these residues are not at levels that may cause harm. For many of these compounds, legal action limits/levels in food have been established or their use is banned. Fera delivers two separate UK surveillance programs: the statutory plan covers > 30,000 samples of UK produce each collected for a different analysis, whereas the non-statutory program is much smaller (1400 samples) and focuses mainly on imports. The later requires multiple tests on each sample for which a multi-residue approach is essential.

We have been using three Xevo TQ-S instruments with UPLC to deliver much of the above programs. Case studies will be presented to show how the enhanced sensitivity and additional capabilities are helping us deliver determination of a range of different classes of analytes and many combined as a multi-residue suite.

V-4. Monday Lunchtime, July 18, 2011 12:15 p.m. to 1:15 p.m. Bruker Corporation
Location: Horizon Rooms

A Compound-based Approach to Simplify Method Development and Data Processing for Multi-residue Analysis by GC-MS/MS

Kefei Wang; Ed John George; Patrick Jeanville; Steven Schachterle; Meredith Conoley; Bruker Corporation, Chemical and Applied Markets Division, 3500 W Warren Ave, Fremont, CA 94538 USA

GC-triple quadrupole mass spectrometer (GC-MS/MS) in multiple reaction monitoring (MRM) mode has become the method of choice for targeted screening of multi-residues due to its ability to monitor many product ions of a large number of compounds. However, method development, including both setting up the hundreds to thousand MRMs for data acquisition and assigning each MRM to its analyte for data processing, is complicated and time consuming. In this study, a simplified compound-based approach to MRM method development is demonstrated for multi-residue pesticide (>250) analysis on a new GC-MS/MS system. The MRM acquisition method can be built by selecting compounds from factory-provided compound library that groups all MRMs under their compound's name, and the data processing method will be automatic generated based on the acquisition method. The new approach also implements a timed compound acquisition for MRM dwell time optimization and eliminates the need for chromatographic segmentation.

V-5. Tuesday Breakfast, July 19, 2011 7:15 a.m. to 8:15 a.m. LECO Corporation
Location: Horizon Rooms

Looking for "Things in Stuff" or "Stuff in Things" - Opportunities for the Complementary Nature of High Performance Time of Flight Mass Spectrometry and High Performance GC (GCxGC) TOF-MS in the Analysis of Pesticides and Other Contaminants in Complex Matrices

Lucas Smith, Joe Binkley, Jeff Patrick; LECO Corporation; St. Joseph, Michigan

This seminar will examine the application of high performance, mass spectrometry-based analytical technologies to the detection and quantitation of pesticides, herbicides and other compounds in complex matrices. High performance GC will be evaluated as two-dimensional GC (GCxGC) with time-of-flight mass spectrometric detection, and high performance MS will use a novel configuration of time-of-flight MS in the context of LC and GC interfaced to a multi-reflecting time-of-flight instrument which offers exceptional resolving power (100,000 for LC and 50,000 for GC) and mass accuracy (< 1 ppm). Analytes will be determined in complex matrices including water samples, wine, animal feed, and fruit/vegetable matter. The comprehensive but complementary nature of high performance mass spectrometry and high performance GCxGC analyses will be discussed. The benefits of high resolving power in mass spectrometry will be compared and contrasted to the high resolution offered by GCxGC in addressing different analytical problems. These considerations will include targeted versus non-targeted analysis, sensitivity, quantifiability, and matrix complexity, among others. The capabilities of high performance mass spectrometry in LCMS analysis will be demonstrated with an emphasis on how the attributes of resolving power and mass accuracy benefit analyses for both quantitation and analyte identification.

V-6. Tuesday Lunchtime, July 19, 2011 12:15 p.m. to 1:15 p.m. UCT, Inc.
Location: Horizon Rooms

Chemical Implications of QuEChERS

Mike Telepchak, UCT, 2731 Bartram Rd., Bristol, PA 19007; mtelepchak@unitedchem.com

SPE, dispersive SPE, Solid Phase dispersion, filtration SPE or whatever else you would like to call it still depends on the same chemical principles. If you wish to get the best performance out of the technique, you need to understand the chemistry.

This presentation will discuss the mechanisms encountered in SPE and how the chemistry affects the different mechanisms.

Since C18 is the most popular phase in environmental analysis, we will discuss the chemistry of C18 columns and how the nature of the bonded phase is affected by the manufacturing process.

The nature of more selective mechanisms, such as ion-exchange, will also be discussed and the affect of pH and distribution coefficients will be included.

V-7. Wednesday Breakfast, July 20, 2011 7:15 a.m. to 8:15 a.m. AB SCIEX
Location: Horizon Rooms

Advanced Tools to Screen for Unknown and Non-targeted Contaminants 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.

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. The AB SCIEX TripleTOF™ 5600 LC/MS/MS system is capable of performing targeted and non-targeted screening in a single LC-MS/MS run. Highly sensitive MS spectra were measured with unmatched speed, high resolution and mass accuracy allowing accurate and reproducible quantitation of targeted compounds at lowest concentration levels. High resolution and accurate mass MS/MS spectra were used to additionally confirm the identity based on mass spectral library searching. The acquired full scan MS and MS/MS data can further be used to retrospectively mine data for non-targeted compounds.

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-8. Wednesday Lunchtime, July 20, 2011 12:15 p.m. to 1:15 p.m. Agilent
Location: Horizon Rooms

Expanding Lab Capability using Agilent's GC/QQQ Analyzer with a New Versatile 1000-compound Pesticide MRM Database
Kai Meng, Agilent Technologies, 2850 Centerville Road, Wilmington, DE, 19808; 302-636-8388, chin_meng@agilent.com

GC/Tandem Quadrupole provides excellent sensitivity and selectivity in analyzing complex matrices. The capability of the analysis in a lab, however, is largely determined by the completeness of the MS/MS transitions in an acquisition method. Agilent Technologies recently develops a new MS/MS transition pesticide database of 1000 compounds to address many of the limitations most labs are facing.

This new MS/MS transition Database:
• allows users to build acquisition methods without acquiring expensive or hard-to-get pesticide standards

• includes exact retention times for either constant flow or constant pressure method; RI (retention index) values are also included for easy migration to other GC oven programs
• has multiple MS/MS transitions (average 7) for each compound

The data acquisition method can be easily created from a subset of MS/MS transitions selected from the database.

Screening and Identification of Non-targeted Compounds with Confidence Using an Exact Mass Spectral LC-MS/MS Library

Jerry Zweigenbaum, Agilent Technologies, Inc., 2850 Centerville Road, Wilmington, DE 19808, 302-636-3661, j_zweigenbaum@agilent.com

Pesticide and xenobiotic screening of non-targeted compounds is increasingly being performed by full spectrum accurate mass measurement mass spectrometers. This presentation will describe a database with over 7500 compounds including pesticides, toxins and pharmaceuticals containing MS/MS spectra for over 2500 compounds collected at 3 collision energies. The effectiveness of searching LC/Q-TOF MS and MS/MS data for food samples extracted with QuEChERS with this database and library will be discussed along with strategies for the experimental settings to collect the data.

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• Regulatory issues
• Pesticide residue monitoring
• Risk assessment issues

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MEETING PROGRAM

Sunday, July 17, 2011

8:30 am-4:30 pm	**Short Course** - Robert Voyksner **Understanding and Optimization of LC/MS/MS to Develop Successful Methods for Identification and Quantitation in Complex Mixtures**	Snowy Egret
5:00 – 8:00 pm	Registration	Grand Palm Colonnade
4:30 – 5:30 pm	Volunteers Orientation	Grand Palm Colonnade
5:30 -10:00 pm	Exhibitor Setup	Pavilion
6:00 – 6:30 pm	Moderator Training	Long/Bird/Indian Key Ballrooms
6:00 pm →	Technical Session Setup	Long/Bird/Indian Key Ballrooms
6:00 pm →	Poster Board Set Up	Pavilion
6:30 – 7:30 pm V-1	**Thermo Scientific Seminar** James Chang, Thermo Fisher Scientific **Non-Target Screening with High Resolution Bench Top Orbitrap (Exactive)/SIEVE**	Banyan & Citrus Rooms
8:00 – 9:00 pm V-2	**Restek Evening Seminar** Jack Cochran, Julie Kowalski, Sharon Lupo, Michelle Misselwitz, Amanda Rigdon-Restek Corporation, Bellefonte, Pa; and Frank Dorman, The Pennsylvania State University, University Park, PA **High Quality Analysis of Pesticides in Marijuana for Food and Medicine using QuEChERS, Cartridge SPE Cleanup, GCxGC-TOFMS, and LC-MS/MS.**	Banyan & Citrus Rooms

Monday, July 18, 2011

All Day	Registration	Grand Palm Colonnade
11:00 am →	Exhibition & Posters	Pavilion
7:15 – 8:15 am	Early Morning Coffee	Pavilion and Grand Palm Colonnade
7:15 – 8:15 am V-3	**Waters Breakfast Seminar** Simon Hird, Head of LC-MS/MS Team, Food and Environment Research Agency (FERA), Sand Hutton, York, North Yorkshire, UK **Use of the Xevo TQ-S as Applied to the Routine and Not So Routine Analysis of Animal Tissues for Residues of Veterinary Medicines**	Horizon Rooms
8:30 – 11:00 am	**Opening Oral Session** Session Chairs, Alaa Kamel and Lynda Podhorniak	
8:30 -8:50	**FLAG Works/President Organizing committee**	
8:50 -9:15 O-1	**Steven Bradbury** –Director of EPA Office of Pesticide Programs, USA ***Priorities, Challenges and Vision of the US EPA Office of Pesticide Programs***	
9:20 -9:45 O-2	**Ionara Pizzutti,** André de Kok -Federal University of Santa Maria- Brazil ***Comprehensive Integrated Multiresidue Analysis of Pesticides and Mycotoxins: Development, Optimization, Validation and Implementation in Routine Analysis***	

9:50-10:15 O-3
Chensheng (Alex) Lu, **Frank Schenck**, retired FDA, Atlanta, GA, Jon Wong – Harvard School of Public Health, Department of Environmental Health, USA
Longitudinal Food Consumption Frequency Determination and Its Implication for Dietary Pesticide Exposure and Risk Assessment

10:20-10:40 O-4
Carl Winter, University of California, Davis, USA
Pesticide Residues in Imported, Organic, and "Suspect" Foods: Using Good Science to Understand Risks

10:40-11:00 O-5
Carl Winter, University of California, Davis, USA
Stayin' Alive: A Musical Look at Contemporary Food Safety Issues

11:00 – noon
Exhibition Opening Pavilion

11:00 – noon
Poster Session A: Gulf BP Oil Spill Effects on Food & Water, Pharmaceuticals & Veterinary Drugs, and International Monitoring Programs
Session A authors at posters
Paul Yang and Lawrence Zintek co-coordinators of poster sessions

12:15 – 1:15 pm V-4
Bruker Corporation Lunch Seminar **Horizon Rooms**
Kefei Wang; Ed John George; Patrick Jeanville; Steven Schachterle; Meredith Conoley, Bruker Corporation, Chemical and Applied Markets Division, Fremont, CA
A Compound-based Approach to Simplify Method Development and Data Processing for Multi-residue Analysis by GC-MS/MS

1:30 – 3:05 pm
Gulf BP Oil Spill Effects on Food & Water
Session Chair, Jo Marie Cook

1:30-1:50 O-6
Gina Ylitalo et al., – NOAA Fisheries, Northwest Fisheries Science Center, USA
Ensuring Seafood Safety After the Deepwater Horizon Oil Spill

1:55-2:15 O-7
Jo Marie Cook, Walter Hammack, Jason Stepp and Roy Jenkins – Florida Department of Agriculture and Consumer Services, USA
Florida's Response to the Gulf Oil Spill

2:20-2:40 O-8
Katerina Mastovska, Wendy R. Sorenson, Jana Hajslova, Covance Laboratories Inc., USA
Status of AOAC Collaborative Study on PAHs in Seafood

2:45-3:05 O-9
Yoko Johnson- Minnesota Department of Agriculture, USA
Analysis of PAHs in Seafoods by QuEChERS Based Extraction and Triple Quadrupole GC-MS

3:10 – 3:55 pm
BREAK for Exhibition & Posters Pavilion

3:55 – 5:05 pm
Pharmaceuticals and Veterinary Drug Residues
Session Chair, Sherri Turnipseed

3:55-4:15 O-10
Perry Martos, Fiona Jayasundara, University of Guelph, Canada
Veterinary Drug Residues from the Perspective of Method Development and Regulatory Compliance

4:20-4:40 **Suil Kang** - United Nation University, S. Korea
O-11 ***Pharmaceuticals and Personal Care Products in Surface Water and Waste Water in Ho Chi Minh City, Vietnam – Occurrence and Typical Range in 2010***

4:45-5:05 **Steven Lehotay**, Alan R. Lightfield, Lucia Geis-Asteggiante, Marilyn J. Schneider
O-12 Agricultural Research Service, USDA, USA
What is the Current Best Method for Multiclass, Multiresidue Analysis of Veterinary Drug Residues in Animal Tissues?

5:30 pm	Depart for Salvador Dali Museum	TradeWinds Hotel Entrance
9:30 pm	Return from Dali Museum to Hotel	Museum where Buses parked

Tuesday, July 19, 2011

All Day Registration, Exhibition & Poster Session

7:15 – 8:15 am Early Morning Coffee in Exhibit Hall (Pavilion) and Grand Palm Colonnade

7:15 – 8:15 am **LECO Breakfast Seminar** Horizon Rooms
V-5 Lucas Smith, Joe Binkley, Jeff Patrick, LECO Corporation, St. Joseph, MI
Looking for "Things in Stuff" or "Stuff in Things" - Opportunities for the Complementary Nature of High Performance Time of Flight Mass Spectrometry and High Performance GC (GCxGC) TOF-MS in the Analysis of Pesticides and Other Contaminants in Complex Matrices

8:30 – 10:30 am **Global Harmonization of Residues**
Session Chair, Andrè de Kok

8:30-8:50 **Mike Doherty** –EPA Office of Pesticide Programs, USA
O-13 ***Residue Chemistry – A Global Regulatory and Risk Assessment Perspective***

8:55-9:15 **Volker Bornemann** – Smithers Viscient, LLC, USA
O-14 ***Efforts in Residue Chemistry Harmonization. A Crop Protection Industry Perspective***

9:20-9:40 **Johannes Corley,** D. L. Kunkel, J. J. Baron – IR-4 Rutgers University, USA
O-15 ***JMPR Submissions and Establishing Codex MRLs for Specialty Crops, Their Importance in Global Trade***

9:45-10:05 **Salah Soliman** –Alexandria University, Egypt
O-16 ***Harmonization of Pesticide MRLs and Registration: A Global Initiative***

10:10-10:30 **Jane Steward** – BASF, USA
O-17 ***OECD Harmonized MRL Calculator***

10:30 – noon **Exhibition & Posters** Pavilion

11:00 – noon **Poster Session B: Advances in Analytical Methods for Chemical Contaminants**
Session B authors at posters
Paul Yang and Lawrence Zintek co-coordinators of poster sessions

12:15 – 1:15 pm	**UCT, Inc. Lunch Seminar**	Horizon Rooms
V-6	Michael J. Telepchak, UCT, Bristol, PA **Chemical Implications of QuEChERS**	
1:30 – 3:05 pm	**International Monitoring Programs** Session Chair, Katerina Mastovska	
1:30-1:50 O-18	**Daniela Brocca** – European Food Safety Authority (EFSA), Italy ***Pesticide Residues in Food - Monitoring Programs in Europe***	
1:55-2:15 O-19	Carazo,E., Acuña,J., Masís,M., Calderón, E., Castro, M., **Jessie Matarrita,** Vargas,E., Alfaro, P. CICA, University of Costa Rica ***Monitoring Pesticide Runoff to the Costa Rican Caribbean Sea***	
2:20-2:40 O-20	**Paul Yang,** Shahram Tabe, Serei Thach and Rajesh Seth – Ontario Ministry of the Environment, Canada ***Detection of Pharmaceutically Active Compounds in the Detroit River Watershed***	
2:45-3:05 O-21	**Jamie Ellis** – University of Florida, USA ***Pesticide Effects on Honey Bees: Myth or Reality***	
3:10 – 4:00 pm	**BREAK** for Exhibition & Posters	Pavilion
4:00 – 5:30 pm	**Forum on Mass Spectrometry** (Moderator, W. Hammack)	
5:30 - 6:30 pm	**Organizing Committee Meeting**	
7:00 - 8:00 pm	Reception by TradeWinds	Pavilion
8:00 pm →	Night Beach Volleyball Game	On the Beach

Wednesday, July 20, 2011

Until noon	Registration, Exhibition & Poster Session	
7:15 – 8:15 am	Early Morning Coffee in Exhibit Hall (Pavilion) and Grand Palm Colonnade	
7:15 – 8:15 am	**AB Sciex Breakfast Seminar**	Horizon Rooms
V-7	André Schreiber, AB SCIEX, Concord, Ontario, Canada **Advanced Tools to Screen for Unknown and Non-targeted Contaminants in Food Samples**	
8:30 – 9:40 am	**Oral Session: Advances in Analytical Methods for Chemical Contaminants** Session Chair, Chuck Stafford	
8:30-8:50 O-22	**Jana Hajslova**, Adam Vavrous, Tomas Cajka, Jana Pulkrabova- Institute of Chemical Technology, Czech Republic ***A Novel Solution for the Rapid Control of Multiple Pesticide Residues in Tea***	

8:55-9:15 O-23 **Jon Wong**, Kai Zhang, Paul Yang, Douglas G. Hayward, James Chang, Center for Food Safety & Applied Nutrition, FDA, USA
Optimization and Application of Ultrahigh Performance Liquid Chromatography-Single Stage Orbitrap Mass Spectrometry for Screening Pesticides in Foods

9:20-9:40 O-24 **Robert Trengove,** Bruce Peebles, Katherine Rousetty- Murdoch University, Australia
Minimising False Positives and False Negatives in Residue Screening of Grains and Oil Seeds: Use of Matrix Matched Standards and Their Impact on LODs

9:45 – 11:00 am **BREAK** for Exhibition & Posters Pavilion
Final Votes by the Attendees for Best Poster are Due by Noon in the Ballot Box

11:00 – 12:10 pm **Oral Session: Advances in Analytical Methods for Chemical Contaminants** *(continued)*
Session Chair, Chuck Stafford

11:00-11:20 O-25 **Kazuaki Iijima** – The Institute of Environmental Toxicology, Ibaraki, Japan
Lessons Learned at IET Analytical Laboratory in Wake of Japan's Huge Earthquake

11:25-11:45 O-26 **Tom Gould** – Bayer, USA
Conversion of Three Old Style Analytical Methods to LC/MS-MS Technology Utilizing Isotopic Internal Standards

11:50-12:10 O-27 **Jim Stry** – Dupont Crop Protection, USA
Trends in Residue Method Development at DuPont Crop Protection

12:20 – 1:20 pm V-8 **Agilent Lunch Seminar** Horizon Rooms
Kai Meng, Agilent Technologies, Delaware
Expanding Lab Capability using Agilent's GC/QQQ Analyzer with a New Versatile 1000-compound Pesticide MRM Database

Jerry Zweigenbaum, Agilent Technologies, Inc., Delaware
Screening and Identification of Non-targeted Compounds with Confidence Using an Exact Mass Spectral LC-MS/MS Library

1:30 – 4:35 pm **Oral Session: Advances in Analytical Methods for Chemical Contaminants** *(continued)*
Session Chair, Steven Lehotay

1:30-1:50 O-28 **Tuija Pihlström** – National Food Administration, Sweden
Analysis of Pesticide Residues by Means of the Swedish Ethyl Acetate Method (SweEt)

1:55-2:15 O-29 **Sara Edison**, Lora A. Lin, Lenin Parrales, FDA, Forensic Chemistry Center, USA
Practical Considerations for Rapid Screening for Pesticides Using Ambient Pressure Desorption Ionization with High-Resolution Mass Spectrometry

2:20 2:40 pm O-30 **Gae Ho Lee**, Jun Lee, Dong Wook Lee, Young Woong Ahn, So Yeon Kim, Jong Eun Kwon- Chung Nam National University, S. Korea
Rapid Determination of Multi-Residues in High Fat Content Samples by GC/MS and LC-MS/MS Using Liquid–Liquid Extraction Followed by Centrifugation, Freezing and Dispersive Solid Phase Extraction as Cleanup Steps

2:45-3:00 pm	**BREAK**	Grand Colonnade
3:00-3:20 O-31	**Kai Zhang**, Jon Wong Center for Food Safety & Applied Nutrition, FDA, USA ***Protocols for the Collection of an Electro-spray Ionization Mass Spectral Library – Development, Intra- and Inter-laboratory Validation and Identification of Unknown in Food and Environmental Samples***	
3:25-3:45 O-32	**Abdou Mekebri**- California Deptartment of Fish and Game, USA ***Recent Advances in the Analysis of Pyrethroid Insecticides in Surface Water and Sediments by Tandem GC/MS***	
3:50-4:10 O-33	**Lucia Pareja**- Universidad de la República, Uruguay ***Development and Application of Multiresidue Methods for the Analysis of Post-Emergence Herbicides within the Rice Cropping Ecosystem***	
4:15-4:35 pm O-34	**Steven F. Volker**, Dana T. Lujan, National Wildlife Research Center, USA ***Determination of Anticoagulant Pesticide Residues in Water, Soil and Terrestrial Crab***	
4:40 – 5:00 pm	**Poster Awards and Closing**	

Meeting Adjourns

Mark your calendar for next year's workshop, July 15-18, 2012



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POSTERS

Session A (Posters numbered P1- P57)
Gulf BP Oil Spill Effects on Food & Water, Pharmaceuticals & Veterinary Drugs, and International Monitoring Programs
Authors stand by their posters from 11:00 am – noon & 3:10 -3:55 pm on Monday

Session B (Posters numbered P58- P111)
Advances in Analytical Methods for Chemical Contaminants
Authors stand by their posters from 11:00 am-noon & 3:10-4:00 pm on Tuesday & 9:45 -11:00 am on Wednesday

P-1 **A Rapid LC/MS Method for Determination of Teflubenzuron in Salmon Tissue**
Narong Chamkasem et al.; FDA, Atlanta, GA

P-2 **Validation of a Method for the Analysis of Veterinary Drug Residues in Frog Legs and other Aquacultured Species using Quadrupole Time-of-Flight LC-MS**
Sherri Turnipseed et al.; US Food and Drug Administration, Denver, CO

P-3 **Effective Sample Preparation For Multi-Residue LC-MS Determination of Veterinary Drugs in Meat and Milk**
Michael S. Young and Kim van Tran; Waters Corporation, Milford, MA

P-4 **Residue Depletion of Tritium-Labeled Ivermectin in Yellow Perch Following Oral Administration**
Donglei Yu et al.; US Food and Drug Administration, Laurel, MD

P-5 **A Dispersive SPE using a New High Capacity Lipid Removal Material or a C18: Analysis of Veterinary Drugs In The Samples Of Animal Origin by LC-MS-MS**
Olga Shimelis et al.; Sigma-Aldrich, State College, PA

P-6 **Using Statistics for Validation of Calibration Certificates**
Paulo Alexandre de Toledo Alves and Valdemar Luiz Tornisielo University of São Paulo - USP, Piracicaba, São Paulo, Brazil

P-7 **Exposure Assessment of the Korean Children to Neonicotinoid Pesticides through Fruiting Vegetable Consumption**
Min-Kyoung Paik et al.; Rural Development Administration, Suwon, Kyeonggido, Republic of Korea

P-8 **Rapid Screening and Quantitation of Multi-Class Antibiotics in Milk using Multiplexed Online SPE-LC-MS/MS**
Stacy Tremintin; AB SCIEX, Foster City, CA, USA

P-9 **Determination of the Penicillin G Antibiotic In Natural Waters by Applying a Dairy Farm Amperometric Biosensor for Analysis and Validation**
Diana Catalina Rodriguezand Gustavo Peñuela; Univesity of Antioquia, Medellin, Antioquia Colombia

P-10 **Simultaneous Determination of Nitrofuran Metabolites and Chloramphenicol in Shrimp by LC-MS/MS with a Single Extraction Procedure**
Aref M. El-Demerdash et al.; US Food and Drug Administration, Lenexa, KS

P-11 **Molecularly Imprinted Polymers for SPE of Nitroimidazole Antibiotics from Tissue and Liquid samples**
Elena Gairloch and Ecevit Yilmaz; Biotage, Charlotte, NC

P-12 **Establishing Multi-Class Drug Residue Analysis Methods to Meet Laboratory, Regulatory, and Customer Needs: The eCAL Perspective**
Lauryn Bailey et al.; Eurofins Central Analytical Laboratories, Metairie, LA

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Mary Blackburn et al.; Thermo Fisher Scientific, San Jose, CA

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Michelle Misselwitz et al.; Restek Corporation, Bellefonte, PA

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Jonathan Pelt and Amy Brown; Florida Dept. of Agriculture's Chemical Residue Laboratory, Tallahassee, FL

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Isabel Cristina Sales Fontes Jardim and Liane Maldaner; University of Campinas - UNICAMP, Campinas, São Paulo Brazil

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Kenneth J. Rosnack et al.; Waters Corporation, Milford, MA

P-55 **Pesticide monitoring to meet regulatory requirements by the direct injection of drinking water**
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Joan Stevens and Andy Zhai; Agilent Technologies Inc, Wilmington, DE

P-58 **Screening Method for 30 Pesticides in Green Tea Extract Using Automated Online Sample Preparation with LC-MS/MS**
Yang Shi; Thermo Fisher Scientific, Franklin, MA

P-59 **A Turn-key Solution for Automated Detection of Organic Contaminants in Food Matrices and Economic Adulteration**
James Chang; Thermo Scientific, San Jose, CA

P-60 **Analysis of 400+ Pesticides in a Single Run Using Triple Quadrupole Mass Spectrometer**
Jia Wang et al.; Thermo Fisher Scientific, San Jose, CA

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Maciej Bromirski et al.; Thermo Fisher Scientific, Bremen, Germany

P-62 **Quantitation of Organophosphate Insecticides in Drinking Water Using Automated Online Sample Preparation and a 3-D Ion Trap**
Mary Blackburn et al.; Thermo Fisher Scientific, San Jose, CA

P-63 **Utilization of High Resolution LC-MS for Screening and Quantitative Analysis of Pesticides in Food Matrix using a Q-Exactive Bench Top Orbitrap Platform**
Jia Wang et al.; Thermo Fisher Scientific, San Jose, CA

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David Steiniger et al.; Thermo Scientific, Austin, TX

P-65 **Determination of organochlorine pesticides in carrots by QuEChERS and GC-ECD**
Valentina Domingues et al.; Requimte, ISEP, Porto, Portugal

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Diego Alejandro Ahumada Forigua; et al.; Centro de Bio-Sistemas. Universidad Jorge Tadeo Lozano, Bogotá D.C., Cundinamarca, Colombia

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Andrea del Pilar Mojica Cortés et al.; Universidad Nacional de Colombia, Bogota, Cundinamarca Colombia

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Lucia Pareja et al.; Universidad de la República, Montevideo, Uruguay.

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Eugene Chang et al.; Food and Drug Administration, Irvine, CA

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Brian Musselman et al.; IonSense, Inc., Saugus, MA

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Jerry Zweigenbaum; Agilent Technologies, Inc., Wilmington, DE

P-73 **Analysis of Fluorinated Alkyl Compounds in Bio-solid Matrix by GC Q-TOF Mass Spectrometry**
Anthony Macherone; Agilent Technologies, Inc., Wilmington, DE

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Chris Sandy et al.; Agilent Technologies UK Ltd, Winnersh, Berkshire, UK

P-75 **Analysis of Trace Level Pesticides in Fruits and Vegetables by GC/MS/MS Using Agilent Ultra Inert Liners with Wool**
Limian Zhao and Kai Meng; Agilent Technologies Inc., Wilmington, DE

P-76 **Analysis of Pesticide Residues in Rice Using QuEChERS AOAC Kit by LC-MS/MS Detection**
Joan Stevens; et al.; Agilent Technologies Inc, Wilmington, DE

P-77 **High Mass Accuracy Provides Enhanced Selectivity for the GC/MS Analysis of Pesticides in Food**
Philip L. Wylie et al.; Agilent Technologies, Wilmington, DE

P-78 **US EPA UPLC/MS/MS Method Development for Analytes of Concern**
Lawrence Zintek et al.; US EPA, Chicago, IL

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Lawrence Zintek; US EPA, Chicago, IL

P-80 **LC-MS Library System Protocol and Possible Future**
Lawrence Zintek; US EPA, Chicago, IL

P-81 **Rapid Screening of Grain Samples for Seed Treatment Pesticides Utilizing a Waters ASAP (Atmospheric Solids Analysis Probe) and Xevo Mass Spectrometer**
Mike Roscoe et al.; Canadian Grain Commission, Winnipeg, MB Canada

P-82 **Determination of 64 Organochlorine, Organonitrogen and Organophosphorus Pesticides in Olive Oil by Optimized QuEChERS Procedure**
Xiaoyan Wang et al.; UCT, Inc., Bristol, PA

P-83 **Determination of Seven Problematic Pesticides in Olive Oil**
Xiaoyan Wang et al.; UCT, Inc., Bristol, PA

P-84 **Automated QuEChERS Extraction for the Determination of Pesticide Residues in Foods using Gas Chromatography/ Mass Spectrometry**
Edward Pfannkoch et al.; GERSTEL, Inc., Linthicum, MD

P-85 **Screening and Quantitation of pesticide residues in food using LC-MS/MS and Identification using MS/MS Library Searching**
YunYun Zou et al.; AB SCIEX, Concord, ON Canada

P-86 **Non-Targeted Screening for Food Residues using High Resolution Accurate Mass LC-MS/MS and Statistical Data Processing**
Andre Schreiber et al.; AB SCIEX, Concord, Ontario Canada

P-87 **Determination of Pesticide Residues in Vegetable Juices using the Triple Quadrupole LC-MS/MS and GC-MS/MS, and a Single Quadrupole Mass Spectrometer GC-MS**
Rima Juskelis et al.; IFSH, Bedford Park, IL

P-88 **Food Testing made Simple and Reliable – Fully Automated and Integrated Modular QuEChERS Sample Preparation**
Alejandro Perez and Annie Schnyder; Chemspeed Technologies, Augst, BL Switzerland

P-89 **High Throughput Screening of Food Contact Materials**
Kenneth J. Rosnack et al.; Waters Corporation, Milford, MA

P-90 **QuEChERS Approach for Determination of Seven Pesticides Residues in Quinoa (Chenopodium quinoa willd) Using GC-µECD**
Franz Zirena Vilca; et al.; Center of Nuclear Energy in Agriculture CENA. University of Sao Paulo, Piracicaba, São, Paulo, Brazil

P-91 **QuEChERS Approach for Determination of Seven Pesticides Residues in Honey Using GC-µECD**
Franz Zirena Vilca; et al.; Center of Nuclear Energy in Agriculture CENA. University of Sao Paulo, Piracicaba, São Paulo, Brazil

P-92 **QuEChERS Approach for Determination of Pesticides Residues in Strawberries Using GC-µECD**
Paulo Alexandre De Toledo Alves et al.; Center of Nuclear Energy in Agriculture CENA. University of Sao Paulo, Piracicaba, São Paulo, Brazil

P-93 **Rapid Analysis of Pesticide Residues using DART-MS**
Tomas Cajka et al.; Institute of Chemical Technology, Prague, Czech Republic

P-94 **Developing a Multi-residue Pesticide Method for Testing Fatty Foods**
Ryan Picou et al.; Eurofins Central Analytical Labs, Metairie, LA

P-95 **Method Improvements for the Analysis of Glufosinate in Plant Matrices**
Ian Murphy; Bayer CropScience, Stilwell, KS

P-96 **A LC/MS/MS Method for the Detection of Penflufen Residues in an Aquatic Dissipation Study using Rice SeedsTreated with Penflufen**
Derek Netzband et al.; Bayer CropScience, Stilwell, KS

P-97 **Fast Screening of Pesticides and Toxins Using Multiple Analytical Techniques at the Food and Drug Administration**
Chris Sack; et al.; U. S. Food and Drug Administration, Lenexa, KS

P-98 **Comparison of 4 Ethyl Acetate Based Methods and Original QuEChERS for the Analysis of Pesticide Residues in Blueberries**
Natalia Besil et al.; Facultad de Química, Universidad de la República, Montevideo, Montevideo Uruguay

P-99 **Automated LC/MS Analyses of Pesticides and Mycotoxins Using QuEChERS and DPX Tips**
William E. Brewer et al.; University of South Carolina, Columbia, SC

P-100 **Screening for Pesticides by LC/MSMS and GC/MSMS in Muscle Tissue**
Catalina Yee et al.; USDA, Alameda, CA

P-101 **Large Volume Splitless Injection Using an Unmodified Splitless Injection Port for the Analysis of Halogenated Pesticides and Brominated Flame Retardants**
Michelle Misselwitz et al.; Restek Corporation, Bellefonte, PA

P-102 **Method for the Analysis of 4-methylimidazole by LC/MS/MS**
Julie Kowalski et al.; Restek Corporation, Bellefonte, PA

P-103 **Comprehensive Screening for PAHs in Sugarcane Products after QuEChERS with Sweet Separations using an Rxi-17Sil MS column, GC-MS, and GCxGC-TOFMS**
Julie Kowalski et al.; Restek Corporation, Bellefonte, PA

P-104 Pesticide Analysis for Organic Cut Roses using QuEChERS, GC-TOFMS, and GCxGC-TOFMS
Jack Cochran et al.; Restek Corporation, Bellefonte, PA

P-105 The QuEChERS Tablet
Jack Cochran et al.; Restek Corporation, Bellefonte, PA

P-106 A Comprehensive Approach to Pesticide Residue Monitoring, Including Non-target Analysis, for Fruits, Vegetables, and Nuts, Using QuEChERS, LC-MS/MS, and GCxGC-TOFMS
Jack Cochran et al.; Restek Corporation, Bellefonte, PA

P-107 Evaluation of Elution Solvents for Three Solid-Phase Extractions to Predict Appropriate Clean-up Method by Matrix and log P Characteristics of Pesticides
Guk-Tak Han et al.; National Agriculture Products Quality Management Service, Daejeon, Chung-Nam, Republic of Korea

P-108 Analysis of Amitraz and 2,4-Dimethylaniline Residues in Colombian Honey By UFLC-MS
Julio César España Amórtegui et al.; Laboratorio de análisis de residuos de plaguicidas- Universidad Nacional deColombia, Bogotá, Cundinamarca, Colombia

P-109 Analysis of Pesticides in Celery Extract by UHPLC-TOF-MS
Jason Weisenseel et al.; PerkinElmer, Shelton, CT

P-110 One Step Extraction, Clean-up and concentration of Chlorinated Pesticides in Raw Coffee Beans
Tom Hall and Jim Westmooreland; FMS, Inc., Watertown, MA

P-111 Modern Water Analysis:The use of Modern Chromatography coupled to In-Line Solid Phase Extraction (SPE) / ESI –Triple Quadrupole / Hybrid Linear Ion Trap Mass Spectrometry, for the Analysis of Surface, Drinking and Wastewater
Frederic Ciner and Ed Dabrea, Jupiter Analytix, Jupiter, FL

ORAL PRESENTATION ABSTRACTS

O-1 Priorities, Challenges and Vision of the US EPA Office of Pesticide Programs

Steven Bradbury, Director of **U.S. EPA**, Office of Pesticide Programs, 1200 Pennsylvania Ave., NW, Washington, DC 20460; 703-305-7090, bradury.steven@epa.gov

The role of EPA is to protect human health and the environment. The EPA Office of Pesticide Programs (OPP) depends uponaccurate and defensible scientific data to help inform our regulatory decisions and assure safe pesticide use. This presentation will focus on the different ways OPP uses laboratory analytical data, from pre-registration scientific review through post-registration compliance monitoring and enforcement. The presentation will also discuss current OPP challenges and the vision needed to address these challenges in the coming years.

O-2 Comprehensive Integrated Muti-Residue Analysis of Pesticides and Mycotoxins in Food Products: Development, Optimization, Validation and Implementation in Routine Analysis

Iona Regina Pizzutti, André de Kok**, Federal University of Santa Maria** (UFSM) - Center of Research and Analysis of Residues and Contaminants (CEPARC), UFSM- Chemistry Department, Av. Roraima 1000, Bldg. 18, office 2215, Campus, Camobi, Santa Maria, **Brazil,** 97105-900; 55 55 3220 9458, ionara.pizzutti@ceparc.com.br, pizzutti@quimica.ufsm.br

In order to safeguard consumer health, monitoring and enforcement of pesticides residues and other contaminants is of utmost importance. Low default limits of 0.01 mg/kg for pesticides and even lower for some mycotoxins are set by the European Union. Official control and private laboratories are putted under increasing pressure to offer analytical services with fast result delivery times at lower costs using analytical methods meeting the requirements of the low reporting limits. This is typically achieved by using multi-residue methods able to include hundreds of analytes in one generic extraction method. Including analytes from different classes of contaminants for the same type of food, further increases efficiency, not only of the analytical methods but also of the sampling costs. In this presentation, the development, optimization and validation of an integrated sample preparation and analysis method for 229 pesticides and 36 mycotoxins in food products will be presented. UPLC-MS/MS was applied for two separate, short, optimized runs for pesticides and mycotoxins. Various GC-MS (/MS) detection methods (Triple-quad/ITD) and modes (EI/NCI) were critically compared for the analysis of GC amenable pesticides. Practical application in routine analysis has shown its benefits and some typical survey results.

O-3 Longitudinal Food Consumption Frequency Determination and Its Implication for Dietary Pesticide Exposure and Risk Assessment

Chensheng (Alex) Lu[3], Frank Schenck[1], Jon Wong[2]

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3.Chensheng (Alex) Lu, Mark and Catherine Winkler, Associate Professor of Exposure Biology Department of Environmental Health, **Harvard School of Public Health**, Landmark Center West, Room 404N, 401 Park Drive, P.O. Box 15677, Boston, MA 02215 617-998-8811, cslu@hsph.harvard.edu

In response to the calls for more direct measurements of pesticide residues in foods that infants and children consumed, as well as the impact of regulatory agencies' actions on assessing dietary pesticide risks, this data represents continuing efforts in monitoring pesticide residues in foods. We measured pesticide residues in 24-hour duplicate food samples collected from a group of forty-six young children participating in the Children's Pesticide Exposure Study (CPES). Parents were instructed to collect 24-hr duplicate food samples of all conventional fruits, vegetables, and fruit juices equal to the quantity consumed by their children, similarly pre-washed/prepared, and from the same source or batch. Individual or composite food items were analyzed for organophosphates (OP) and pyrethroid insecticides residues. We collected a total of 239 24-hr duplicate food samples collected from the forty-six CPES children. We found 19% of those food samples contained at least one OP or pyrethroid insecticides. A total of eleven OP insecticides, levels ranging from 1 to 387 ng/g, and three pyrethroid insecticides, levels ranging from 2 to 1,133 ng/g, were measured in children's food samples. We found many of the food items consumed by the CPES children were also on the list of the most-contaminated food commodities reported by other organization. The frequent consumption of food commodities with episodic presence of pesticide residues that are suspected to cause developmental and neurological effects in young children supports the need for further mitigation.

O-4 Pesticide Residues in Imported, Organic, and "Suspect" Foods: Using Good Science to Understand Risks

Carl Winter, **University of California**, Food Science and Technology, Davis, CA 95616; 530-752-5448, ckwinter@ucdavis.edu

Consumers are frequently warned about the potential risks from exposure to pesticides in their foods. Concerns have frequently been voiced about consuming imported produce and eating conventional forms of the "Dirty Dozen" commodities identified by the Environmental Working Group as posing the greatest threats to consumers. Risk assessments are presented that compare the risks from consumption of imported vs. domestic fruits and vegetables and that quantify exposure levels to the most frequently detected pesticides on the "Dirty Dozen"list. The risks from pesticide residues in organic foods are also discussed.

O-5 Stayin' Alive: A Musical Look at Contemporary Food Safety Issues

Carl Winter, **University of California**, Food Science and Technology, Davis, CA 95616; 530-752-5448, ckwinter@ucdavis.edu

Join the "Elvis of E. coli" as he provides musical parodies designed to entertain and inform about current food safety issues. Be prepared to sing along!

O-6 Ensuring Seafood Safety after the Deepwater Horizon Oil Spill

Gina Ylitalo, Bernadita Anulacion, Jennie Bolton, Daryle Boyd, Richard Boyer, Douglas Burrows, Jon Buzitis, Denis da Silva, David Herman, Jonelle Herman, Margaret Krahn, Ronald Pearce, William Reichert, Catherine Sloan, Karen Tilbury, Walton Dickhoff, and John Stein; **NOAA Fisheries**, Northwest Fisheries Science Center, 2725 Montlake Boulevard East, Seattle, WA 98112; 206-860-3325, gina.ylitalo@noaa.gov

The Deepwater Horizon Oil Spill in April 2010, released approximately 200 million gallons of crude oil into the Gulf of Mexico. Thus, there was concern about the risk to human health through consumption of contaminated seafood. Federal and state agencies have worked together to ensure that seafood from the Gulf is safe to eat. Our analytical facility has analyzed more than 3100 seafood specimens for selected polycyclic aromatic hydrocarbons (PAHs) using a comprehensive gas chromatography/mass spectrometry method. FDA and NOAA worked together to develop and validate rapid methods that accurately and precisely measure PAHs and dispersants associated with the spill. Although chemical compounds associated with spill have been detected in seafood collected in federal waters, the concentrations were much lower than FDA levels of concern for human health effects and thus did not pose any appreciable risk to human consumers of seafood products from the Gulf.

O-7 Florida's Response to the Gulf Oil Spill

Jo Marie Cook, Walter Hammack, Jason Stepp and Roy Jenkins
Florida Department of Agriculture and Consumer Services, 3125 Conner Blvd., Lab #3, Tallahassee, FL 32399-1650; 850-617-7505, jomarie.cook@freshfromflorida.com

Working with FDA and FERN, Florida's Bureau of Chemical Residue developed capabilities to screen for polycyclic aromatic hydrocarbons, first by the NOAA GC/MS method and then by the FDA's LC Fluorescence method. Recently, the laboratory also added FDA's LC/MS method for the analysis of dispersants to their seafood screens. The methods, analytical results and lessons learned in responding to emergencies will be discussed.

O-8 Status of the AOAC study on PAHs in seafood

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2. **Covance Laboratories Inc**., 3301 Kinsman Boulevard, Madison, WI 53704
3. **Institute of Chemical Technology**, Prague, Faculty of Food and Biochemical Technology, Department of Food Chemistry and Analysis, Technická 3, 166 28 Prague 6 **Czech Republic**

As a response to the 2010 oil spill in the Gulf of Mexico, the AOAC International formed a stakeholder panel on seafood contaminants and issued a call for methods for the analysis of polycyclic aromatic hydrocarbons (PAHs) in seafood. For a collaborative study, the panel selected a GC-MS based method developed within a European integrated project CONffIDENCE by the Institute of Chemical Technology (Prague, Czech Republic) for the determination of PAHs, polychlorinated biphenyls (PCBs) and polybrominated diphenyl ethers (PBDEs) in fish and seafood. Nineteen important PAHs and their relevant alkyl homologues were included in the collaborative study, with mussel, oyster and shrimp being the tested matrices. This presentation will provide status of the study and discuss issues encountered during its course.

O-9 Analysis of PAHs in Seafoods by QuEChERS Based Extraction and Triple Quadrupole GC-MS

Yoko S. Johnson, **Minnesota Department of Agriculture**, 601 Robert Street North, St. Paul, MN 55155; 651-201-6534, yoko.johnson@state.mn.us

An analysis of PAHs in seafood using QuEChERS based extraction and GCMSMS was developed and validated by recovery experiments in 4 seafood matrices – crab, finfish, oyster, and shrimp. The extraction employs QuEChERS with dispersive SPE using clean-up sorbent that includes C18. The acetonitrile extract is partitioned into hexane and analyzed by GCMSMS. The procedure was validated by spike recovery experiments of 15 parent PAHs and 5 alkylated homologs. Recoveries are 67-130%. The LOQ for B(a)P are 1.0 -2.8 ppb depending on matrix. For all 15 parent PAHs, the LOQs are less than 10% of the FDA's concerned level. Method accuracy was also evaluated by analyzing NIST Standard Reference Material (SRM) 1974b. Ten of the 14 PAHs fell within the NOAA control limits.

O-10 Veterinary Drug Residues from the Perspective of Method Development and Regulatory Compliance

Perry Martos and Fiona Jayasundara**, University of Guelph**, 95 Stone Rd. W., Guelph, Ontario N1H8J7, **Canada**; 519 767-6209, pmartos@uoguelph.ca

The study of veterinary drug residues in foods of animal origin is ongoing and in demand of methods that provide comprehensive analysis for more compounds, and classes, in a rapid and cost effective manner. Risk assessment can also be a simultaneous

objective with data generated from a regulatory program, which requires the development of methods employing the best available technologies, such as LC-MS/MS and LC-qTOF. This work focuses on the on-going use of a recently developed multi-residue, multi-class veterinary drug method and its application in a rapid throughput regulatory environment. Challenges with the method are highlighted, particularly as the requirements and demands increase to accurately analyze more samples with the same resources and the need to retain method continuity for the purposes of longitudinal data comparisons.

O-11 Pharmaceuticals and Personal Care Products in Surface Water and Waste Water in Ho Chi Minh City, Vietnam – Occurrence and Typical Range in 2010

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Many pharmaceuticals and personal care products (PPCPs) have been consistently found in the aquatic environment such as surface waters and wastewater effluents at the ppt (ng/L) to ppb (µg/L) levels. Their occurrence and contamination are of recent environmental concerns because some have biological effects in living tissues and organisms, may likely to be bioaccumulative and very little is known about their collaborative effects. We present in this talk initial monitoring results obtained from the 2010 PPCP typical background study using samples collected from canals and rivers around and in Ho Chi Minh City (HCMC), Vietnam. A total of 62 PPCP target compounds were monitored using automated solid phase extraction (SPE) for sample preparation and liquid chromatography/tandem mass spectrometry in two different laboratories. Quality assurance data obtained during the analysis, as well as results of inter-laboratory comparison of analytical standards will be discussed. Occurrence and concentration of target analytes analyzed will be discussed in terms of temporal and seasonal trends.

O-12 What is the Current Best Method for Multiclass, Multiresidue Analysis of Veterinary Drug Residues in Animal Tissues?

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Multiclass, multiresidue methods (MMMs) have been commonly used in pesticide monitoring programs for more than 45 years, but this concept was not commonly considered for veterinary drug residue monitoring until recently when LC-MS became widely accepted and available in regulatory labs. MMMs are beneficial over single analyte or single class methods because they generally increase monitoring scope and greatly improve laboratory efficiency (higher sample throughput and less labor and costs). In our lab, we evaluated 6 MMMs from the scientific literature for UHPLC-MS/MS analysis of more than 60 high priority veterinary drug analytes in beef kidney. The methods include those from Mol et al., Martos et al., Lehotay et al., Leepipatpiboon et al., Stubbings et al., and Kaufmann et al. (with small modifications of each). A main conclusion is that all the methods achieved acceptable results for qualitative screening identification purposes below U.S. tolerance levels. In terms of quantification (integrated peaks normalized to isotopically-labeled sulfamethazine), each method gave between 70-120% recoveries with <20% RSDs (18 replicates at 3 levels) for about 70% of the analytes in spiked samples. Only minor differences were observed in terms of quality of results, matrix effects, and sample throughput (except the Kaufmann et al. method was much slower and more laborious). We compared results of incurred samples to devise and validate a new streamlined method to achieve the most broadly acceptable results in the fastest time, which still entails sample cleanup to maintain instrument ruggedness.

O-13 Residue Chemistry – A Global Regulatory and Risk Assessment Perspective

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In a world of increasing global trade and marketing, and decreasing resources, the need for harmonization of pesticide data requirements and regulatory levels has become all the time more important. The recent adoption by OECD member countries of harmonized guideline and guidance documents for pesticide residue chemistry studies is a major step forward in promoting global harmonization. The framework of studies that make up the suite of pesticide residue data requirements creates a foundation for making regulatory decisions and conducting human health risk assessments, with the overarching goal being the maintenance of a safe and healthful food supply. This framework will be discussed, with an emphasis on the role of analytical methods, which underpin many of the residue studies.

O-14 Efforts in Residue Chemistry Harmonization - An Industry Perspective

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Within the crop protection industry many changes have occurred over the past two decades with considerable consolidation of companies and rationalization of product lines. At the same time, the regulatory hurdles have significantly increased, making the development of new products with a favourable regulatory profile increasingly difficult, expensive, time consuming and economically riskier. New products are typically developed for global markets and for uses in several geographic regions. However, non-harmonized guidelines and data requirements as well as differences in data evaluation and regulatory policies between individual national regulatory authorities lead to additional development costs, obstacles to work sharing between regulatory authorities, and trade barriers for internationally traded food (and feed) commodities. In conclusion, the currently ongoing regulatory harmonization efforts of FAO, WHO, CCPR, and in particular, those of the OECD Residue Chemistry Expert Group (RCEG), are supported strongly and actively by CropLife International.

O-15 JMPR Submissions and Establishing Codex MRLs for Specialty Crops, their Importance in Global Trade

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Over the last two decades the agrochemical industry has focused on developing reduced risk, greener chemistries and products for major crops. In the U.S. the IR-4 project has been developing data to facilitate registrations of these products on specialty crops. However, grower's use of these new products has been limited because maximum residue limits (MRL) supporting export commodities is not always available. Since 2008, IR-4 has been instrumental in collecting and submitting data suitable for supporting Codex MRLs to the Joint FAO/WHO Meetings on Pesticide Residues (JMPR), a body that provides independent scientific expert advice to the Codex Alimentarius Commission and its Committee on Pesticide Residues. These submissions have resulted in the establishment of a number of Codex MRLs. Acceptance of these Codex MRLs by regulatory authorities and their harmonization with country specific MRLs is important in lowering trade barriers and promoting use of reduced risk and safer chemistries globally in agriculture. This presentation will include a discussion of suitability of data for JMPR submissions, the submission process, and recommendations for acceptance and harmonization of MRLs.

O-16 Harmonization of Pesticide MRLs and Registration: A Global Initiative

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Every year new pesticides are introduced to the global market. Before these new products are granted approval, distributed, and made available to users they must be rigorously assessed by national governments to ensure that they meet current health, environment and safety standards. Older pesticides already on the market are also re-evaluated to guarantee that they continue meeting these standards. This process of pesticide regulatory review and registration is mostly carried out by each country and hence is often duplicative and resource-intensive. Since the late 1980s, many countries have agreed to work jointly through bilateral or multilateral programs to harmonize approaches to pesticide registrations and maximum residue level (MRL) standard setting. This process is critical in order to help governments work jointly to evaluate the risks of individual pesticides more quickly and thoroughly, and ensure the smooth flow of global trade. These "work-share" programs allow experts from collaborating countries, and sometimes from invited countries, to discuss, share and combine their knowledge, expertise, methods, findings and processes. As an outcome, these governments are able to approve the introduction of much safer, effective products to the market and replace more hazardous ones more quickly and efficiently. This also results in an increase of the number of pesticide products that are available to specialty crop growers worldwide. This has encouraged us to call for a global initiative to harmonize registration of pesticides worldwide. This initiative, if adopted, will also facilitate the global harmonization of pesticide MRLs by supporting Codex as the common standard. With the cooperation of the USDA, two preliminary meetings related to this initiative were held in Nairobi, Kenya and in Alexandria, Egypt. Two other meetings were organized in collaboration with the African Union. Both were held in Addis Ababa, Ethiopia, where issues of harmonization of pesticide registration and MRLs were addressed. Furthermore, a multinational field study to test similarity in measured residues of a pesticide on/in tomato was performed in 22 worldwide distributed locations. This study was supervised by the USDA and its results will help throwing light on possibility of harmonization of Pesticide MRLs. Analysis of perception and importance of this initiative will be discussed in this conference. The methodology used to clarify its benefits and the experimental method used to worldwide standardize measuring residues of a pesticide on/in a crop will be presented.

O-17 OECD Harmonized MRL Calculator

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Until very recently, individual countries and organizations had different approaches to calculating MRLs from underlying residue data. This disparity was one of the major deterrents in the effort to harmonize MRLs globally. In the Spring of 2008, to promote international harmonization for the process of establishing MRLs, the OECD Residue Chemistry Expert Group (RCEG) charged a technical working group with the challenge of developing a new procedure for calculating MRLs that would harmonize the existing EU and NAFTA MRL procedures. The new MRL procedure, embodied in an automated spreadsheet referred to as the OECD MRL Calculator, was released by the OECD in March 2011 for use throughout the world. The methodology and performance of the OECD MRL Calculator will be described and demonstrated with typical field trial residue data. Challenges associated with the harmonization of the statistical and non-statistical "regulatory" aspects of MRL setting procedures will also be discussed.

O-18 Pesticide Residues in Food - Monitoring Programs in Europe

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In Europe, National and EU coordinated programs are in place to monitor pesticide residues in food items. Member States have to report the control results to the European Food Safety Authority (EFSA); these are published in an annual report. The European countries put a lot of efforts in taking samples (ca. 70,000 on a yearly basis) and analyzing pesticide residues (approximately 800 substances). The annual report provides an overview on the pesticide residues in food observed by 29 countries (27 EU Member States and 2 EFTA countries), reports the rate of sample compliance with the legal limits and assesses the exposure of consumers through their diets. In 2010, the EU countries collected and submitted control data according to a new model, which allows both data providers and recipients the full description of the samples and analytical parameters for evaluation purposes and more accurate exposure assessment. These improvements will also help better inform and support risk managers in regulating the safe use of pesticides.

O-19 Monitoring Pesticide Runoff to the Costa Rican Caribbean Sea

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The monitoring coastal component of the GEF-funded Reducing Pesticides Run-off to the Caribbean Sea (Costa Rica, Colombia and Nicaragua) Project (GEF-(REPCar)), being executed by the UNEP's Caribbean Environment Programme has been implemented in Costa Rica by the Centro de Investigación en Contaminación Ambiental (CICA) of the Universidad de Costa Rica (UCR) in collaboration with the Centro de Investigación en Ciencias del Mar y Limnología (CIMAR-UCR) and the Ministerio del Ambiente, Energía y Telecomunicaciones of Costa Rica (MINAET). The project began in 2006 and is scheduled to end this year, 2011. The coastal monitoring component is comprised of two phases. During the first phase, baseline information of pesticides runoff to the Caribbean was established through the monitoring of the main watersheds that drain into the Caribbean Sea and one monitoring campaign in which marine sediment was collected. The objective of the second phase was to establish a monitoring programme to determine and characterize the pesticide contamination level of six important watersheds which were chosen based on the results of the first phase. The second phase involved the conventional sampling techniques which were combined with passive samplers, semi-permeable membrane devices (SPMD) and polar-organic chemical-integrative samplers (POCIS). Also in two watersheds, the flow was measured in order to calculate the pesticide loads and biomonitoring was applied using macroinvertebrates as bioindicators.

O-20 Detection of Pharmaceutically Active Compounds in the Detroit River Watershed

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In an extensive study, the persistence of a number of pharmaceutically active compounds (PhACs) in the Detroit River watershed were investigated using both liquid chromatography/tandem mass spectrometry and LC/high resolution mass spectrometry. The study took place over a period of one year and covered the transport path of target substances from the effluent of a local sewage treatment plant (STP) to the intake of a water treatment plant (WTP). It was found that a large portion of these pollutants with significant concentrations and frequencies of detection at the effluent of the STP became undetectable or significantly less frequent at the intake of the WTP. Natural processes such as dilution, adsorption onto suspended solids, sedimentation, and degradation were counted for the reduction in the concentrations of these compounds. However, the concentrations of a number of the pollutants were still above the detection limit at the intake of the WTP. The observations were explained by chemical and physical properties of the target substances as well as the water quality parameters. In this paper, the results of the study will be presented and comparison will be made to previous studies in the same area and other water sources around Ontario and Canada.

O-21 Pesticide Effects on Honey Bees: Myth or Reality

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The number of managed honey bee colonies in the U.S. has declined since the 1940's. In 2006, the decline accelerated due to a phenomenon called Colony Collapse Disorder (CCD). The cause of CCD remains under investigation but many scientists and beekeepers suggest that pesticide exposure may contribute to the losses. The average honey bee can forage 3-5 miles from the nest. Consequently, a single colony can forage in an area ranging from ~28 – 80 miles2, thus being exposed to a range of chemical toxins in the environment. Through surveys for pesticides in bee colonies, investigators found 121 different pesticides and metabolites in sampled colonies. The average colony contained residues from 6 different pesticides, some of those from chemical treatments used in colonies by beekeepers. New data on pesticide effects on bees show that pesticides are capable of impacting adult and immature bees chronically, possibly explaining at least some colony losses.

O-22 A Novel Solution for the Rapid Control of Multiple Pesticide Residues in Tea

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Current analytical approaches used for the determination of pesticide residues in complex matrices with a low content of water such as tea are time consuming, require high consumption of solvents, and only a limited number of analytes is involved. During the isolation step troublesome bulky coextracts (e.g., caffeine) might be co-isolated. When not removed from the extracts, these compounds can cause serious problems during both gas chromatographic separation and mass spectrometric detection. Therefore, streamlining of sample preparation strategy for this type of matrix is an essential part of their analysis. In addition, detection step is also crucial to avoid potential interferences with target masses of particular analytes. In this study, we present a novel solution for the rapid control of GC-amenable multiple pesticide residues (>100) in tea. Both sample preparation and detection steps were optimized with the expectation of enhanced speed, high accuracy, and improved selectivity.

O-23 Optimization and Application of Ultrahigh Performance Liquid Chromatography-single stage Orbitrap Mass Spectrometry for Screening Pesticides in Foods

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The use of ultrahigh pressure liquid chromatography- single stage Orbitrap high resolution mass spectrometry (UHPLC-HR-MS) for the multiresidue analysis of pesticides was evaluated. System performance was evaluated based on sensitivity, the reproducibility of area counts obtained from extracted ion chromatogram (XIC), and short term mass axis stability at four different mass resolution and concentrations. Analytical results showed the UHPLC-HR-MS can achieve instrument detection limits (IDL) from the high ppt (ng/kg) to low ppb (µg/kg) for the pesticides studied. Short term mass accuracy of the HR-MS was determined to be < 5 ppm in various matrices including orange, carrot, spinach, flour, and raisin; well within the acceptable limits required for confirmation analysis. At low ppb levels and in complex plant-based matrices, a resolution of at least 50,000 is required for the identification of molecular isobars to differentiate target analytes from interfering artefacts. The ability of UHPLC- HR-MS in identifying pesticide isobars with monoisotopic mass < 50 ppm was demonstrated, allowing the complete identification of 504 pesticides in one 12 min LC- HR-MS run. Analysis of unknowns in incurred samples using LC- HR-MS and statistical post data analysis were also demonstrated as well. By comparing control and treated vegetable samples, we were able to distinguish the samples in terms of the relative abundance of a number of analytes and identify parent pesticides in treated samples using SIEVE 1.2 software and ChemSpider databases.

O-24 Minimising False Positives and False Negatives in Residue Screening of Grains and Oil Seeds: Use of Matrix Matched Standards and their Impact on LODs

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It is well established that the presence of residual matrix enhances response for some compounds in GC/MS based analysis and if not addressed can lead to false positives. Some compounds show reduced response in the presence of matrix for GC/MS analysis, whilst the complete absence of matrix results in degradation of some compounds leading to false negatives. The impact of matrix matched standards on false positives; false negatives and LODs have been investigated following QuEChERS extraction for wheat, barley and canola using triple quadrupole GC/MS. Examples of compounds which are affected by matrix interference in each grain or oil seed type will be presented and the potential for false positives, false negatives and compromised LODs illustrated. Compound loss through matrix "binding" of residue compounds will be discussed. The relationship between calibration standard format, inlet type and inlet liner activation on residue LOD will be discussed.

O-25 Lessons Learned at IET Analytical Laboratory in Wake of Japan's Huge Earthquake

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Throughout April, Japan was continuously bombarded by aftershocks from the initial earthquake. As time passed, the extent of the damage to farming, fishing, and manufacturing became fully realized. The coastal areas ruined by the tsunami are yet to be cleared. The situation remains serious at the Daiichi nuclear power plant. Our laboratory wishes to pass along our experiences so that we and others may be better prepared for any future natural disasters of this nature such as fall prevention strategy, importance of sub-samples, cooperation and relationships with other laboratories.

O-26 Conversion of Three Gas Chromatography Analytical Methods to LC/MS-MS detection (with help from isotopic internal standards)

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Ethofumesate is a sugar beet herbicide that was historically analyzed by derivatization with detection by GC/FPD. The modified method includes additional analytes, eliminates the derivatization, and detects the analytes by LC/MS-MS. The modified method is more sensitive and reproducible. Pyrimethanil is a fungicide that was historically analyzed by derivatization with GC/ion trap detection. As part of a data call-in, the EPA requested conversion of the detection step to the more reliable LC/MS-MS technique. The method was modified to detect the underivatized analytes by LC/MS-MS. Propineb is a dithiocarbamate fungicide historically analyzed by acidic conversion to carbon disulfide, followed by GC analysis. The acidic hydrolysis was retained, however the remainder of the method was revised to utilize LC/MS-MS detection. This modified method is more sensitive, reproducible, and allows differentiation of propineb residues from ethylene dithiodicarbamates.

O-27 Trends in Residue Method Development at DuPont Crop Protection

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Registrants routinely develop trace level residue and environment methods designed to support regulatory studies and to gain new product registrations. Liquid chromatography coupled to tandem mass spectrometry (LC/MS/MS) is the preferred detector for these methods. Tandem mass spectrometry offers selectivity, low detection limits and the ability to analyze to a wide range of compounds in complex matrices during a single analysis. Instrument manufactures have consistently improved the sensitivity of

triple quadrupole instruments allowing method developers to minimize resource consuming and laborious extractions and extract purification steps, previously required for trace level residue analysis. Most importantly the increase in instrument sensitive has allowed efficient methods to be developed that minimized LC/MS matrix effects, allowing for accurate quantitation of residues while using external standards. Time of Flight (TOF) mass spectrometers play a role in screening and the quantitative analysis of food and environmental residues. The mass resolution and rapid scan times of TOF mass spectrometers offers a clear advantage over triple quadrupole instruments, especially when screening samples for possible unknown contaminants. Moreover, improvements in the linear detection range of TOF instruments have allowed these systems to be used for quantitative analysis. Current method strategies and the relationship to the improvements in LC/MS/MS instruments will be presented. Examples will be discussed which demonstrate how DuPont Crop Protection has implemented improved instrument detection levels to streamline residue method development and sample analysis. Also, the possible role of TOF and QTOF instruments in future residue method development will be addressed.

O-28 Analysis of Pesticide Residues by Means of the Swedish Ethyl Acetate method (SweEt)

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National Food Administration has a tradition of analysing pesticides residues with ethyl acetate since 1989. Present methods are based on extraction with ethyl acetate and determination with GC-MS/MS and LC-MS/MS. The multi residue methods have been revised continuously, resulting in improved and simplified methodology for analysing of pesticide residues. The need of clean up has been eliminated entirely for fruits and vegetables and cereals. To recover basic pesticides in fruits and vegetables, the addition of sodium hydrogen has been used to all matrices. Smaller sample sizes in the methods, earlier 75 g down to 10 g for fruits and vegetables is extracted with 20 ml of ethyl acetate, earlier 200 ml. Furthermore, in improving analysis, the chromatographic LC aspects were taken into consideration including the use of columns with small particles (7µm) resulting in the reduction of analytical time in LC-MS/MS from 30 minutes to16 minutes. Finally, the solvent change from ethyl acetate to methanol has been eliminated saving time and reducing analytical steps. The direct injection of ethyl acetate to LC-MS/MS has shown to separate all analytes selectively. Consequently, the methods has been simplified providing significant benefits in terms not only of sample throughput and improved efficiency but also cost-effective and is environmentally better than the previous methods reducing the volume of organic solvents remarkably. In this presentation, the analytical features, application fields and results will be discussed.

O-29 Practical Considerations for Rapid Screening for Pesticides Using Ambient Pressure Desorption Ionization with High-Resolution Mass Spectrometry

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A rapid screening method for pesticides has been developed to streamline processing of produce entering the United States. Foam swabs were used to recover multiclass mixtures of >100 pesticides from the surfaces of a variety of produce matrices. The swabs were analyzed using direct analysis in real time (DART) ionization coupled with a high resolution Exactive Orbitrap™ mass spectrometer. This study expands the types of commodities analyzed using this method and explores the feasibility of compositing multiple units of produce per batch to analyze a representative sample. The varying topography of the commodities necessitated minor modifications to the method. Additionally, incurred samples that had undergone traditional sample preparation using QuEChERS (quick, easy, cheap, rugged, and safe) were analyzed using both LC-MS and DART-MS and the results from the two techniques were found to be comparable. The use of swabs greatly increases sample throughput by reducing sample preparation and analysis time.

O-30 The Rapid Determination of Multi-Residues in High Fat Content Samples by GCMS and LCMSMS using Liquid–Liquid Extraction Followed by Centrifugation, Freezing and Dispersive Solid Phase Extraction as Cleanup Steps

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A multi-residue method using liquid–liquid extraction (LLE) followed by centrifugation, freezing and dispersive solid phase extraction (dispersive SPE) as clean up steps and GCMS has been developed for the determination of trace levels of 95 pesticides in soybean oil. LLE has been optimized to extract these pesticide residues from soybean oil by studying the effect of different partitions. Experiments were carried out in order to study the efficiency of using centrifugation and freezing steps as well as the used of primary secondary amine (PSA), florisil, graphite carbon black (GCB) and C18 for dispersive SPE on clean up stages to minimize the co-extract fat. The recoveries obtained ranged from 80 to 114% and the relative standard deviation (RSDs) from 2 to 14% for spiking levels of 0.040, 0.080 and 0.160 mg/kg. The limits of quantification (LOQs) of almost all compounds were below the maximum residue limits (MRLs) established by the Korean legislations for soybean oil. The same procedure has been successfully applied to multi-residue analysis of antibiotics and mycotoxins in high fat content samples such as meat and olive oil, respectively.

O-31 Protocols for the Collection of an Electro-spray Ionization Mass Spectral Library-Development, Intra- and Inter-laboratory Validation and Identification of Pesticides in Food and Environmental Samples

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Protocols for the collection of mass library spectra of 240 pesticides from high performance liquid chromatography quadruple linear ion trap hybrid mass spectrometry (LC-qIT-MS) were developed. Enhanced product ion (EPI) spectra were collected for each pesticide in positive ionization mode, at two on-column loadings, 200 and 2,000 pg, and four different collision energies (CE), 20, 35, 50 eV and 35 eV with collision energy spread (CES) 15 eV, resulting an in-house library that contains over 2,000 spectral entries. The library was validated via both intra- and inter-laboratory studies. The intra-laboratory study optimized chromatographic separation, CE, declustering potential (DP) and ion trap fill times used for the library spectra collection and identification based on EPI spectral library search utilizing an information-dependent acquisition (IDA) screening procedure. The IDA procedure uses scheduled multiple reaction monitoring (sMRM) as a survey scan to trigger EPI experiments to collect and store EPI spectra for 240 pesticides from one single LC injection. Using LC retention times, one sMRM transition in the survey scan and spectral Fit value>80 as the identification criterion, an automated forward search algorithm identified >80% of the 240 pesticides fortified into 16 different food matrices at concentrations > 10 ng/g. Various pesticides in incurred samples were identified and quantitated using the LC-sMRM-IDA-EPI procedure and confirmed by liquid chromatography-tandem mass spectrometry (LC-MS/MS) analysis using two sMRM transitions. The library and IDA-EPI procedure were further validated at three laboratories using the same type of qIT-MS systems. The three laboratories consistently identified pesticides in the same blind test samples. Both intra-and inter-laboratory studies showed that the proposed protocols can be used to develop and validate libraries as a viable tool for the identification and confirmation of unknowns in LC-MS analysis.

O-32 Recent Advances in the Analysis of Pyrethroid Insecticides in Surface Water and Sediments by Tandem GC/MS

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Pyrethroid insecticides are of increasing environmental concern because of their widespread use and high aquatic toxicity. Pyrethroids are frequently detected in California in both agricultural and urban areas and more recently pyrethroids have become a concern in other parts of the United States. Recent advances in analytical instrumentation have allowed the development of new analysis methods for determination of pyrethroid insecticides in surface water and sediments. The analytes target list includes seven pyrethroid insecticides the most used in agriculture and structural pest control in California. Water and sediment sample extracts were analyzed using negative chemical ionization tandem mass spectrometry (NCI-MSMS). Method detection limits have been established for water and sediment that are well below the toxicity of the analytes.

O-33 Development and Application of Multiresidue Methods for the Analysis of Post- Emergence Herbicides within the Rice Cropping Ecosystem

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The post-emergence herbicides currently used in the rice cropping ecosystem in Uruguay are; sulfonylureas, bentazone, bispyribac-sodium, cyhalofop-butyl, quinclorac, clomazone, and propanil but no analytical methodology has been developed for the simultaneous determination of them. An optimized HPLC-DAD based determination of these herbicides was combined with specific sample preparation treatments for soil, water and grain that allowed the straightforward determination of their residues in the three matrices. The developed methodologies allowed the monitoring of rice cropping ecosystem for post-emergence herbicides at limits of quantification in the range of 1 to 5 µg/L and 30 to 250 µg/kg for water and grain respectively, whereas for soil samples the recoveries and LOQs are highly dependent on the type of soils. Main positive findings occurred in water and soils from rice lands, particularly clomazone, quinclorac, propanil and less frequently, metsulfuron methyl.

O-34 Determination Of Anticoagulant Pesticide Residues In Water, Soil, And Terrestrial Crabs

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Three extraction procedures are used to determine diphacinone and brodifacoum residues in water, soil, and terrestrial crabs following rodent eradication operations on Cocos Island, Guam. Water samples are acidified and concentrated onto C18 solid-phase extraction disks and eluted with a chloroform/methanol solution containing ammonium hydroxide. Soil, hermit crab (Coenobita perlatus and C. cavipes), and land crab (Cardisoma carnifex) tissues are extracted with chloroform/methanol solutions. A reversed-phase ion-pair HPLC method with UV and fluorescence detection is used to simultaneously quantify both pesticides. The method limit of detection (MLOD) is 0.16 ng/mL, 0.064 ng/mL, 0.027 µg/g, and 0.015 µg/g for diphacinone, and 0.038 ng/mL, 0.032 ng/mL, 0.0030 µg/g, and 0.018 µg/g for brodifacoum in seawater, standing water, soil, and crabs, respectively. Recoveries from fortified controls are 106(±11)%, 99.0(±4.3)%, 90.5(±4.0)%, and 98.4(±9.7)% for diphacinone, and 110(±14)%, 99(±15)%, 98.1(±0.8)%, and 99(±18)% for brodifacoum in seawater, standing water, soil, and crabs, respectively.



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POSTER ABSTRACTS

P-1 A Rapid LC/MS Method for Determination of Teflubenzuron in Salmon Tissue

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This paper describes a simple high-throughput LC/MS method for the determination of Teflubenzuron in salmon tissue. Salmon tissue is shaken with water/acetonitrile mixture in the presence of NaCl and MgSO4. The fish extract was centrifuged and the supernatant was mixed with an equal volume of water, filtered and analyzed by LC/MS. The average recoveries at 1, 10 and 100 ng/g fortification levels are 65.9%, 88.5%, and 101.37% with relative standard deviation of 6.02%, 1.64% and 3.31%, respectively. Detector linearity covers the range of 0.2 to 100 ng/mL, with a correlation coefficient (r) of 0.9995. The estimate Limit of Detection (LOD) which is defined as the standard concentration that can be detected with signal to noise ratio ≥ 3 is 0.03 ng/mL. The estimated Limit of Quantification (LOQ) which is defined as the standard concentration that can be detected with signal to noise ratio ≥ 10 is 0.1 ng/mL. The LOQ level is equivalent to an incurred concentration of Teflubenzuron in Salmon tissue at 0.3 ng/g.

P-2 Validation of a Method for the Analysis of Veterinary Drug Residues in Frog Legs and other Aquacultured Species using Quadrupole Time-of-Flight LC-MS

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A quadrupole time-of-flight LC-MS method was developed and validated for veterinary drug residues in frog legs and other fish. Samples were extracted using a procedure developed for the analysis of fluoroquinolones. The tissue was extracted with acetic acid/acetonitrile, evaporated and reconstituted in mobile phase. A molecular weight cut-off filter was also used. In addition to fluoroquinolones, other residues detected in frog legs samples included trimethoprim, sulfamethoxazole, and chloramphenicol. The method was reoptimized and validated for these additional residues. Screening of residues was accomplished by collecting TOF data and comparing the accurate mass of compounds to a veterinary drug database. Using MS data, a linear response and acceptable recoveries were observed for a majority of these compounds. In addition, MS/MS data were also generated, and the mass accuracy of product ions was compared to theoretical values. The method was successfully applied to other fish species including shrimp, tilapia, and catfish.

P-3 Effective Sample Preparation For Multi-Residue LC-MS Determination of Veterinary Drugs in Meat and Milk

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To ensure food safety, there is a need for multiresidue UPLC-MS methods that can identify and quantify a wide range of veterinary drug residues from many drug classes. Solvent extraction can be effective for many of these compounds in meat and milk. However, highly water soluble drugs such as sulfanilamide and salbutamol may not be well recovered using this approach. If, instead, an aqueous buffer is used for extraction then there is poor recovery of fat soluble compounds such as phenylbutazone and dexamethasone. In this poster we will discuss effective sample preparation to maximize recovery of the widest possible range of veterinary residues in meat or milk. Also, we will present effective SPE cleanup protocols for reduction of matrix effects detrimental to good UPLC-MS performance. Representative compounds from seven classes of veterinary drugs (tetracycline, macrolide, fluoroquinolone, sulfonamide, beta-lactam, steroid and beta-adrenergic) were selected to demonstrate the effectiveness of the methodology.

P-4 Residue Depletion of Tritium-Labeled Ivermectin in Yellow Perch Following Oral Administration

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Residue depletion and metabolism of tritium-labeled ivermectin (3H-IVM) was studied in yellow perch following oral treatment. Yellow perch were dosed with 3H-IVM at the level of 0.1 mg/kg of body weight (~ 8 uCi) in a gel-capsule via stomach tube. At each of the following withdrawal times, 6 fish were sedated with MS-222, euthanatized, bled, scaled and fillets with adhering skin were collected at 1, 3, 7, 14, 21, 28, 42 days. The bile and plasma were also collected during necropsy procedures. The homogenized muscle tissue, bile and plasma samples were analyzed for total radioactive residue (TRR) analysis by Sample Oxidizer. The homogenized tissue was extracted with methanol for residue characterization by high performance liquid chromatography (HPLC). In this study, a new simplified method for TRR analysis in bile and plasma was developed and validated. The analysis of yellow perch samples indicated that highest TRR concentration was obtained in plasma and muscle by post-dose day 1 and in bile by day

3. The maximum TRR of 44, 226 and 3487 ng/mL (ivermectin equivalent concentration) were detected in muscle, plasma and bile, respectively. The half-lives (T1/2) of TRR in bile, plasma, and tissue, which, represents the total of parent and metabolites, are 11.4, 18.7 and 39.4 days, respectively. HPLC analysis of the muscle extract revealed that the parent drug, 3H-IVM, has a T1/2 of 10.2 days in tissue. By post-dose day 21, about 63% of the TRR in the muscle tissue were two unknown metabolites (42% and 21%). Ivermectin was not detectable by post-dose day 42.

P-5 A Dispersive Spe Using a New High Capacity Lipid Removal Material or a C18: Analysis of Veterinary Drugs in The Samples of Animal Origin by LC-MS-MS.

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The concept of dispersive solid phase extraction (dSPE) cleanup (otherwise known as QuEChERS method) for the analysis of contaminants in food samples continues to gain acceptance. The SPE phases that were used as dSPE sorbent to-date included C18 for the removal of lipid components, primary-secondary amine (PSA) for the removal of acids, polar pigments and sugars and graphitized carbon black (GCB) for the removal of pigments such as chlorophyll. Here we demonstrate the use of a new phase with the capacity for the retention of lipids and other hydrophobic compounds. We applied the new lipid-removal phase for the cleanup of kidney and milk samples using dSPE. The results were compared to the sample cleanup using more conventional C18 dSPE phase. In case of dirtier samples, such as kidney, the new phase outperformed C18 for sample cleanup with higher recovery of veterinary drugs and lower amount of ion suppression in the analytes signals. Similar performance for both dSPE phases was observed in case of milk samples.

P-6 Using Statistics for Validation of Calibration Certificates

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In Brazil the GLP (Good Laboratory Practices) and ISO 17.025:2005 Guidelines provided by INMETRO (National Institute of Metrology, Normalization and Industrial Quality) requires validations for Calibrations Certificates of Laboratory Equipments for Pesticides Residues Analysis. This issue is hard to find and sometimes confusing, because not all regulatory auditors and inspectors accept the regular calculations and evaluations about the expiration date of Certificates and need of new calibrations for each equipment. Focusing standardization of validations at Laboratory of Ecotoxicology CENA/USP – Brazil, was developed, validated and approved a SOP (Standard Operational Procedure) using statistics methods as RSD (Relative Standard Deviation), R-Squared Correlation and T-Test for validation of certificates, comparing new calibrations versus old calibrations. The comparisons helped to extend the validity of certificates via critical analysis by QAU (Quality Assurance Unit), or to take actions for calibrations in shorter times, depending on the usage of the equipments (as balances, thermometers and micropipettes).

P-7 Exposure Assessment of the Korean Children to Neonicotinoid Pesticides through Fruiting Vegetable Consumption

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We estimated the exposure assessment of Korean children to five neonicotinoid pesticides (acetamiprid, clothianidin, imidacloprid, thiacloprid, thiamethoxam) using a probabilistic approach. Pesticide residues data in fruiting vegetables (eggplant, pepper, cucumber, tomato, oriental melon, watermelon, squash and strawberry) were obtained from Rural Development Administration for the 2009 monitoring programme in Korea. Total exposure of five neonicotinoid pesticides for Korean infant(1-2 years old) and toddler(3-6 years old) ranged from 0.325 to 0.808 μg/kg/day and from 0.110 to 0.274 μg/kg/day, respectively, at the 95th percentile. This result showed that for dietary risk assessment of all neonicotinoid pesticide residues through fruit vegetable consumption, infant group and toddler group were exposured about 4.4 and 1.8 times higher, respectively, than general population in Korea. However, the %ADI was much below the level of concern for all children groups, signifying that they are considered to be at a health protective level.

P-8 Rapid Screening and Quantitation of Multi-Class Antibiotics in Milk using Multiplexed Online SPE-LC-MS/MS

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Antibiotics are added to dairy cow feed to prevent mastitis, an udder infection. Presence of antibiotic residues in milk can be problematic; human consumption may cause resistance to medicinal antibiotics. EU and US regulatory agencies have set independent levels for the maximum residue limit of antibiotics in milk. Typical approaches to antibiotic detection focus on single classes of antibiotics or require tedious clean-up. Using online solid phase extraction (SPE) liquid chromatography coupled to a triple quadrupole mass spectrometer (LC-MS/MS), nine classes of antibiotics (80 compounds) are screened and quantified. Sample preparation involved protein precipitation and centrifugation. The mass spectrometer was run in multiple reaction monitoring (MRM) mode. Various brands of milk were examined, results show low part-per-billion levels of antibiotic residues quantifiable in milk.

P-9 Determination of the Penicillin G Antibiotic in Natural Waters by Applying a Dairy Farm Amperometric Biosensor for Analysis and Validation

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The presence of antibiotics in the environment, especially in water sources, is a major problem not only because of the serious consequences for human health, but also because the environmental balance is altered due to the emergence of bacterial strains resistant to antibiotics. Penicillin for its part is one of the most commonly used antibiotics to treat bacterial infections, especially in cattle. The determination of antibiotics can be performed using analytical techniques such as chromatography or from screening methods such as those using biosensors. These methods use a biological sensing element coupled with a transducer to produce a signal proportional to the concentration of the analyte. This paper presents the results and the methodology used to validate the analysis of penicillin G in a water matrix using an amperometric biosensor. The results showed a recovery of 87% for natural water samples with three different concentrations of penicillin. The quantification limit obtained was 20 uM.

P-10 Simultaneous Determination of Nitrofuran Metabolites and Chloramphenicol in Shrimp by LC-MS/MS with a Single Extraction Procedure

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A method is developed for the simultaneous determination of nitrofuran metabolites 3-amino-5-morpholinomethyl-2-oxazolidone (AMOZ), 3-amino-2-oxazolidinone (AOZ), 1 aminohydantoin (AH), semicarbazide (SC) and chloramphenicol (CAP) in shrimp with a single extraction procedure. The method uses LC-MS/MS analysis by positive and negatefive switching electrospray ionization (ESI) applying dynamic multiple reaction monitoring. Validation of the method in shrimp was conducted following the FDA criteria for the analysis of veterinary drug residues in foods. The sensitivity for nitrofuran metabolites is better than 0.5 ppb and for chloramphenicol is bettFer than 0.3 ppb which meet the regulatory requirement for these antibiotics.

P-11 Molecularly Imprinted Polymers for SPE of Nitroimidazole Antibiotics from Tissue and Liquid samples

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Nitroimidazole antibiotics are used to treat anaerobic bacterial and parasitic infections in animals. Regulations vary widely from state to state, but often include restrictions on the use of these compounds in food livestock and permitted levels in food for human consumption. The AFFINILUTE MIP Nitroimidazoles was designed to effectively clean up the samples so that even traces of these analytes can be detected. In this poster, we apply novel SPE scouting plates, based on the manner in which 'similar' molecules are able to exploit the intrinsic functionality of the recognition sites on molecularly imprinted polymers. The plates contain 128 unique separation resins, which allows a vast landscape of separation interactions to be screened. One we had a "hit", we were able to cost-effectively create a very selective SPE phase, ideal for extracting nitroimidazoles from milk and eggs with high recoveries , RSD (avg. 95 ± 7%).

P-12 Establishing multi-class drug residue analysis methods to meet laboratory, regulatory, and customer needs: The eCAL perspective

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There is a definite need to establish accurate methods for the quantification of multiple classes of antibiotic drug residues in a single analysis in a variety of matrices, including edible animal tissues (muscle, liver, kidney), aquaculture (fish tissue), and even powders (dried milk, etc.). Multi-class methods are time, money, and waste savers, and can be beneficial when regulatory agencies require certificates of analysis for several drugs spanning many different drug classes. Many studies on this topic have been published, focused on multi-class residue methods in tissue, milk, grains, etc. Here we present our internal development of a multi-class drug residue method, devised from a composite of many excellent published studies, which meets our laboratory's needs, our customers' needs, and the quantification requirements of global regulatory agencies.

P-13 Effect of Sample Size on Determination of Accurate Pesticide Residue Levels in Japanese Agricultural Commodities

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Variabilities of pesticide residue levels based on sample size were estimated by the individual pesticide residue data in apple,

broccoli, cabbage, grape, and sweet pepper commodities. The relative standard deviation (RSD) values from mean of each number (n=2, 4, 8, 16, 32, 64) of randomly-selected samples were defined as the residue variations on each sample size. The coefficients of correlation (γ2) between estimated residue variations and sample weights by the normal power approximation were greater than 0.87 for all of the agricultural commodities. Estimated residue variations calculated by these numerically-derived equations based on the regulatory recommended sample size (weights from 1 to 5 kg, and numbers from 12 to 24 commodities) ranged from 2.1% for spinach to 14.6% for cabbage. Furthermore, predicted percentages of exceeding maximum residue limit (MRL) are calculated from the deriving equation on this study for a sample based on various sampling size, whose mean residue is less than the MRL, however, part of their individual residues are higher than the MRL. These results indicate that the sufficient sample size and double check analysis are important to obtain the accurate pesticide residue levels in raw agricultural commodities.

P-14 Paclobutrazol (PBZ) behaviour in mango pulp analyzed by HPLC-DAD

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Paclobutrazol (PBZ) is a plant growth regulator that controls tree growth by inhibiting gibberellin synthesis, also used to increase production of cereals and seeds of eucalyptus. In Brazil it is widely used in mango cultivation areas. PBZ is most effective when added to soil, that is rapidly absorbed by roots and translocated to aerial parts (stem and leafs). Therefore, the aim of this study was to evaluate the behavior of the plant growth regulator paclobutrazol 14C-PBZ (and its residues/metabolites) applied in vessels of mango plant (Tommy Atkins) and mango pulp. The product presented low potential for leaching in the study in both vessels (in which rainfall occurs). The translocation of PBZ for mango fruits was not evident in this study because it was not detected in the original product mango pulp, analyzed by high performance liquid chromatography coupled to diode array detector (HPLC-DAD).

P-15 US EPA's ERLN Tool Box

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EPA's Environmental Response Laboratory Network (ERLN), which includes the Water Laboratory Alliance (WLA), is a national network of laboratories that provides analytical capability and sufficient capacity to meet project-specific data quality objectives on an as-needed basis. The ERLN integrates capabilities of existing public sector laboratories with accredited private sector laboratories to support environmental responses. To enhance emergency preparedness, EPA has the following tools and resources available: Compendium of Environmental Testing Laboratories (Laboratory Compendium), Portable High-Throughput Integrated Laboratory Identification System (PHILIS), Standardized Analytical Methods (SAM), Web-based Electronic Data Review (WebEDR). Attendees will obtain a basic understanding of these tools, who they are for, and how the resources can assist users.

P-16 Analysis of GC Amenable Pesticides in Soils From American Healthy Homes Survey Sites: Comparison of Two Different Extraction Protocols

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The American Healthy Homes Survey is a collaborative study by the U.S. Department of Housing and Urban Development and the U.S. Environmental Protection Agency that assessed levels of lead, allergens, and insecticides in a randomly selected, nationally representative sample of residential homes. Soil samples taken at a subset of the homes sampled were sent to Wisconsin State Laboratory of Hygiene, where duplicate weighed portions of each sample were fortified by a surrogate standard (fenchlorphos) and extracted by two methods – (1) A sonication method developed by Wisconsin Occupational Health Lab, and (2) an ASE variation of U.S. EPA Method 8270C. These extracts were then analyzed by GC/MS for seven target analytes (diazinon, chlorpyrifos, fipronil, alpha-chlordane, 4,4-DDT, cis-permethrin, and cypermethrin). Pesticides were detected in 8 out of the 10 soils tested, with alpha-chlordane, permethrin, and 4,4-DDT being detected in low ppm range. Overall, the ASE extraction protocol outperformed extraction by sonication.

P-17 Florida's Year 10-11 Pesticide Residue Surveillance

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A summary of the pesticide residue findings of Florida's pesticide residue surveillance program will be presented. Data will include analytes and amounts found as well as commodities with most pesticides and violations.

P-18 How DPX and Micro Luke Works at SCDA to Screen Produce for Pesticide Residue

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Over the last five years the SCDA has been working on a new rapid extraction method called DPX (Dissposable Pipette Extraction) to screen produce and other matrices for Pesticide Residue. We conduct Market Basket Surveys each week in Supermarkets and Farmers Markets all over our state testing for Chemical Residues that may be harmful, therefore, it is important that we have a 24 to 48 hour turn around on our testing. To do that we needed a faster Extraction Method. We compared the DPX to the Luke and the QuECHRS Methods by running samples of incurred Positives and Spiked produce by all three methods. This poster shows the results of this study and how we have switched to this new method to save time and money. We can do twice as many samples for half the cost and detect more compounds at lower levels using Acetone instead of Acetonitrile with the DPX/Micro Luke Method.

P-19 Determination Of Atrazine And Ametrine Herbicides In Surface Water Of The Piracicaba River, São Paulo, Brasil

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Ametrine and atrazine are among the most used herbicides in the cultivation of sugar cane in the region of Piracicaba River, which probably can be contaminated by these molecules. Thus, these two herbicides are being monitored during February 2011 to January 2012. In February, March and May were found atrazine concentrations below the LOQ, differently of April that presented in the points 4 and 5 concentrations of 1.92 and 0.83 µg•L-1 respectively. In February ametrine presented in the points 1 and 3 concentrations of 0.20 and 0.94 µg•L-1 respectively and in March were found 0.27 and 0.24 µg•L-1 in the point 2 e 5 respectively. In May the ametrine presented in point 6 0,24 µg•L-1, since the other point presented concentrations below the LOQ.

P-20 Analysis and Monitoring of Fluopicolide in Agricultural Products

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This study was conducted to investigate the residue of fluopicolide in 17 commodities collected in Korea. Fluopicolide was analyzed by Korea food code, and the method was validated for optimization of analytical conditions. The method contains methanol extraction and solid phase clean-up followed by HPLC-PDA and LC/MS/MS. The limit of detection (LOD) and limit of quantification (LOQ) were 0.007 mg/kg and 0.02 mg/kg, respectively. The calibration curve showed high linearity (R2=0.9998). The recoveries of fortified samples (rice, apple, potato, mushroom, perilla leaves and soybean) were found to be 84.4 ~ 95.2%. We monitored residual level of fluopicolide in commercial agricultural products (10 vegetables, 3 fruits, soybean, potato, rice and mushroom) collected from 9 provinces. No residual fluopicolide was detected in 96 samples.

P-21 Monitoring of Pesticide Residue in Agricultural Produts using GC/MS/MS in Korea

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This monitoring, which was initiated in 2011 to collect data on pesticide residues in agricultural products, has an important purpose of the investigation of of pesticide residue levels in South Korea. We conducted a survey on 17 agricultural products: rice, cabbage, radish, onion, squash, tomato, spinach, carrot, lettuce, perilla leaf, watermelon, apple, pear, strawberry, soybean, potato and oyster mushroom. All 9 provinces of South Korea were included (namely Seoul, Busan, Incheon, Daegu, Ulsan, Daejeon, Gwangju, Changwon, Suwon). The method for simultaneous determination of 183 pesticide residues in agricultural products has been established. The multi-residue analysis of the pesticides in agricultural products involved extraction with acetonitrile, clean-up using solid-phase extraction(SPE) packed with florisil. The final extract was concentrated in a rotatory evaporator at 40℃, then the residue was dissolved with acetone, and analyzed by GC/MS/MS. Total 137 samples were collected and analyzed, 5 samples (3.6%) were found to contain residual pesticides. other samples were found to be free from pesticide residues. 5 agricultural products reported pesticide residues within safe levels. i.e. not exceeding MRLs (maximum residue limits) for Pesticides in Foods, set by the Korea Food Code. Tomato and strawberry were especially affected by pesticide residues, Procymidone and diethofencarb were particularly prevalent.

P-22 Monitoring of Mandipropamid Residues in Agricultural Products of Korea

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Mandipropamid is a new mandelamide-type fungicide to control foliar Oomycete pathogens in vegetables. Mandipropamid was listed to Korea food code in 2006 and allowed to grape, tomato and Chinese cabbage in Korea. While this fungicide has used in recent years, the monitoring data was not reported yet. This study was carried out to analyze for mandipropamid residues in 17 different types of the agricultural products which were most widely consumed in Korea. We selected 1 cereal grains, beans, mushrooms and potatoes, 3 fruits, 10 vegetables for monitoring and purchased samples at wholesales supermarkets located in the area depending on population distribution. This year, we will analyze manidpropamid residues from all 341 samples. Mandipropamid was analyzed using HPLC/PDA by Korea food code method. To evaluate performance of the method, validation experiments were carried out on 6 matrices such as rice, potato, mushroom, apple, sesame leaf and bean. Recoveries of mandipropamid from fortified samples at limit of quantification were 75%~100% and with less than 10% RSD. No mandipropamid residue was detected in 137 samples until now. These results will be used as fundamental database for risk assessment of pesticides.

P-23 Establishment of the Tolerance for Diazinon and Difenoconazole in Rice Straw in Korea

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Tolerance of pesticide residue in feed have been set up 27 pestices for “formula feeds and forages” in Korea. Rice straw is one of the most widely used cattle feed in Korea. However the tolerance of pesticide residues in feed is comprehensive; “formula feeds and forages”. Thus, This study was conducted for the establishment of the pesticide residues tolerance in rice straw. The evaluated pesticides were diazinon and difenoconazole used in rice. This study was conducted according to the FAO guideline. The estimated MRLs(Maximum Residue limits) in feed were carried out using data of rice trials(in Korea). The maximum residues of diazinon and difenoconazole in rice straw at harvest were 3.00 and 0.94 mg/kg, respectively, the MRL proposal in Korea for rice straw were 10 and 5 mg/kg, respectively. The estimated MRL(Maximum Residue Level) in livestock products was carried out using proposed MRLs of rice straw and livestock feeding studies(Draft Assessment Report of EU). Diazinon and difenoconazole were not detected in the treated livestock. Therefore, the proposed MRLs, diazinon 10 mg/kg and difenoconazole 5 mg/kg, were suitable for the tolerance for pesticide residues in rice straw in Korea.

P-24 Monitoring of the Total Type II Pyrethroid Pesticide Residues in Citrus Oils by Liquid Chromatography-Tandem Mass Spectrometric Determination of Their Specific Metabolites

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Pyrethroids are a class of synthetic insecticides that are widely used in household and agricultural applications. A new, highly sensitive analytical method to determine the total type II pyrethroid residues in citrus oils was developed here through the quantitative analysis of the common metabolite 3-phenoxybenzoic acid (3-PBA) or 4-floro-3-phenoxybenzoic acid (4-F-3-PBA). By using a propriety process to convert type II pyrethroids into 3-PBA or 4-F-3-PBA, which are subsequently quantified by LC-MS/MS, the method can measure the total pyrethroid residues down to sub ppb levels. Because citrus oil represents one of the most difficult matrices for 3-PBA conversion, the methods are readily applicable to assess low level contamination of type II pyrethroids in other matrices.

P-25 UHPLC-APPI-MS Method for High-Sensitivity and High-Throughput Analysis of US EPA Sixteen Priority Pollutants Polynuclear Aromatic Hydrocarbons in Oyster

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In response to Gulf of Mexico deepwater horizon oil spill, we develop a QuEChERS and APPI based UHPLC-MS method for analysis of PAHs in oyster. In comparison with the existing HPLC-UVD/FLD and GC or GC-MS based methods, this method offers better selectivity and higher sample throughput. Ten grams of oyster are used for QuEChERS extraction and d-SPE cleanup. The MDLs (6δ S/N=3) are: Naphthalene: 0.023 ppm; Acenaphthylene: 0.084 ppm; Acenaphthene: 0.107 ppm; Fluorene: 0.013 ppm; Phenanthrene: 0.011 ppm; Anthracene: 0.012 ppm; Fluoranthene: 0.009 ppm; Pyrene: 0.010 ppm; Benzo[a]anthracene: 0.013 ppm; Chrysene: 0.013 ppm; Benzo[b]fluoranthene: 0.021 ppm; Benzo[k]fluoranthene: 0.021 ppm; Benzo[a]pyrene: 0.027 ppm; Dibenzo[a,h]anthracene: 0.013 ppm; Benzo[ghi]perylene: 0.057 ppm; Indeno[1,2,3-cd]pyrene: 0.040 ppm. The spike recoveries range from 77% to 110% with %RSD of 0.6-6.7 at spike conc. (Level 1) lower or much lower than the US FDA levels of concern, and 71% to 92% with %RSD of 0.3-4.3 at Level 2 (5x Level 1).

P-26 US EPA LC/MS/MS Method Development for DOSS, EGBE and DPGBE Analysis.

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The development and implementation of a method for determination of dioctyl sulfosuccinate (DOSS) in sea water by direct injection using liquid chromatography (LC) and detection with tandem mass spectrometry (MS/MS) will be explained. Several hundred samples were analyzed by the US EPA Region 5 and 6 labs during the summer of 2010. DOSS was a major component of dispersants applied to the 2010 Deepwater Horizon Oil Spill. DOSS has also been used as a major component of certain laxatives and numerous products, such as cosmetics and foods. Method for Determination of Dipropylene Glycol Monobutyl Ether (DPGBE) and Ethylene Glycol Monobutyl Ether (EGBE) in sea water by direct injection using liquid chromatography (LC) and detection with tandem mass spectrometry (MS/MS) will be explained. DPGBE and EGBE have a variety of residential and industrial applications such as, cleaning formulations, oil dispersants, surface coatings, inks and cosmetics. These analytes may be released into the environment at levels that may be harmful to aquatic life.

P-27 LC/MS/MS Analysis of PAH's, their Derivatives, and 3 Oil Dispersants in Sea Water

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There have recently been a number of maritime crude oil spills in various parts of the world. Oil spills contribute to release of polycyclic aromatic hydrocarbons and related compounds into the aquatic environment. The presence of PAH's in water and sea foods are of great concern, as some of them have been identified as carcinogenic, mutagenic and teratogenic. These PAH's and their alkyl derivatives have a number of isomers, some of which are toxic, while others are biologically inert so accurate characterization is important. Efforts to ameliorate spills include the use of various dispersants to remove oil slicks and residual oil. In this study, we focused on 3 dispersant compounds, 2-butoxyethanol, dipropylene glycol monobutyl ether and dioctylsulfosuccinic acid, as well as many of the commonly monitored PAH's and their alkyl derivatives. PAH's are traditionally analyzed by GC or GC-MS. However, their analytical run time is quite long because their isomers need to be chromatographically separated for proper analysis. In this study we worked towards separation of these isomers by HPLC with a reasonable run time with good sensitivity.

P-28 Ensuring Seafood Safety with Rapid Screening for Polyaromatic Hydrocarbons Using LC-Fluorescence

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Major oil spills such as the Exxon Valdez in 1989 and the recent Gulf of Mexico spill of April, 2010 have raised concerns over the quality of seafood harvested from these regions. Fish, crustaceans and mollusks may come into contact or ingest the oil thereby introducing potential health risks to consumers. PolyAromatic Hydrocarbons (PAHs) are an important subset of compounds present in oil that have been classified as priority pollutants. The US FDA has established levels of concern ranging from 0.035 mg/kg Benzo(a) pyrene in finfish to 2000 mg/kg combined phenanthrene and anthracene in oysters. To prevent consumption of contaminated seafood and minimize the impact on the seafood industry, a fast screening method is required to analyze these compounds of concern at the stated levels. Here we demonstrate that, following a simple extraction using the Waters DisQuE Dispersive Sample Preparation Kit (QuEChERS), an analysis of PAHs can be achieved in less than 4 minutes using the ACQUITY® H-class with fluorescence detection. Results show that dispersive sample preparation provides a fast and effective method for extracting PAHs from different seafood matrices. The method provides advantages over other sample preparation techniques as accurate results can be achieved with less sample preparation and in a shorter time. With sample preparation times reduced, a rapid chromatographic separation is critical to manage the samples, standards and QCs generated using this approach. The ACQUITY H-Class separation, which was achieved in less than 4minutes, is able to address this demand. This solution allows laboratories to screen for PAHs in seafood, providing results in a timely and economical manner so consumers can be confident that these products are safe.

P-29 Determination of PAH in Seafood: Optimized Sample Preparation Procedures for LC-Fluorescence Screening and GC-MS(MS) Confirmation

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PAH (polycyclic aromatic hydrocarbons) are common in nature both as natural products and from anthropogenic sources. PAH are produced from combustion processes and from coal gasification or coke production and occur naturally as constituents of coal and petroleum. The recent major oil spill in the Gulf of Mexico has focused attention on the problem of PAH contamination of the environment and also on the analytical challenges related to PAH analysis of food, sediment, soil and water. In this presentation we will discuss an optimized sample preparation protocol for determination of PAH in shellfish samples. The initial sample extraction is performed using a QUECHERS method in which a sample is extracted with acetonitrile in the presence of an excess of buffer salts. This technique provides a convenient extract suitable for LC with fluorescence (LC-FL) or selective UV detection with no further workup required. However, the resulting acetonitrile extract is not ideal for GC-MS analysis. A simple, straightforward SPE strategy

will be demonstrated for effective PAH confirmation analysis by GC-MS in oyster and shrimp samples prepared using the QUECHERS approach. Thus, the same QUECHERS extract can be used for both LC-FL and for GC-MS (after cleanup). Also, tandem GC-MS will be compared with single quad GC-MS for the confirmatory PAH analysis.

P-30 Fully Automated Extraction of PAHs From Seawater

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Polycyclic aromatic hydrocarbons (PAHs) are widespread pollutants that can be released into a wide variety of environmental matrices by many different natural and artificial sources. The 2010 Gulf Oil Spill has increased the need for continuous monitoring of PAHs in seawater. Analysis can be time-consuming as low concentrations require large-volume samples, and multi-step manual methods require constant operator intervention.

The J2 Scientific PrepLinc LVi system fully automates the extraction of PAHs from seawater samples by conditioning the SPE sorbent, loading the sample onto the cartridge or disk, and eluting directly to the AccuVap module for concentration and transfer to a GC vial for analysis. A single PrepLinc LVi system can process up to 21 1-liter samples unattended. Recovery data is presented for the EPA's 16 priority pollutant PAHs in salt water matrices using this fully-automated method.

P-31 Fully Automated PAH Analysis in Gulf Seafood

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The 2010 Gulf Oil Spill has increased concerns regarding PAHs in fish and shellfish, and because there is incomplete knowledge concerning long-term affects and the rates at which these materials are absorbed, continued analysis of seafood for PAHs is imperative. Gel Permeation Chromatography (GPC) is a standard cleanup technique for fish tissues, but GPC alone is not always adequate to remove all of the high molecular weight compounds from particularly high-lipid samples, and cannot always remove all compounds that interfere with the analytes in the determinative method. Solid Phase Extraction (SPE) can be used to remove these additional interferences, but additional cleanup can be labor-intensive and increases the risk of analyte loss with each step. This presentation shows how the J2 Scientific PrepLinc system combines GPC and SPE cleanup in a single method to thoroughly and efficiently prepare seafood samples of current concern due to the gulf oil spill.

P-32 Photodegradation of Iprodione at Lab and Pilot Scale: Effect of Classical Parameters

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The main causes of water contamination are industrial discharges and use of pesticides. The most recent water decontamination processes concern the oxidation of organic pollutants. These methods (advanced oxidation processes - AOPs) rely on the formation of highly reactive chemical species that degrade the more recalcitrant molecules into biodegradable compounds. In this work the TiO2 photo degradation of iprodione (IPR) in water, at lab and pilot scale, was studied. The TiO2 concentration (0.5-2 g L-1) and pH (3-9) were optimized. pH 7.27 and 1.35 g L-1 of TiO2 favoured the efficiency of the process. Under optimal conditions the evolution of the substrate, COD, DOC, toxicity were evaluated. In the pilot scale tests (127 L), using direct sunlight, 11 mg L-1 of IPR in a commercial formulation was eliminated after 180 min; while after 360 min of treatment 83% of toxicity (1/E50), 80% of COD and 80% of DOC were eliminated.

P-33 Photo-Fenton Degradation of Carbofuran. Evaluation of the Toxicity and Analysis of Organic By-Products

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Cultivation of plants for economical purposes requires a constant struggle against losses from pests promoted by weeds, insects, diseases. The most frequent agents used for this objective are pesticides. Therefore, in recent times the removal of organic harmful pollutants present in water supplies is investigated by means of a variety of chemical procedures. Among them, photo-Fenton-reaction(Ma, Sung et al. 2010). In this work the degradation of Carbofuran (CBF) in water, was studied. The Fe2+ concentration (0.5-5 mg L-1) and H2O2 (2-50 mg L-1) were optimized at pH 3. As optimization result, 5 mg L-1 of Fe2+ and 34.55 mg L-1 of H2O2 favoured the process. Under optimal conditions the evolution of the substrate, COD, DOC, toxicity were evaluated. Ma, Y.-S., C.-F. Sung, et al. (2010). "Degradation of carbofuran in aqueous solution by ultrasound and Fenton processes: Effect of system parameters and kinetic study." Journal of Hazardous Materials 178(1-3): 320-325.

P-34 Application of EPA Method 8261A to Reduce Measurement Bias: A Comparison Study Between Purge & Trap, Static Headspace, & Vacuum Distillation Techniques for VOC Analysis

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For decades, Method 8260 from EPA's Test Methods for Evaluating Solid Waste (SW-846) has been used for analysis of volatile organic compounds (VOCs) in environmental samples. However, this method can produce biased measurements of VOCs in some sample types due to partitioning into water-immiscible phases and/or differences in ionic strength between the samples and the calibration standards, and these matrix effects are poorly compensated by the internal standards. SW-846 method 8261 is an alternative calibration model that uses the trend in recovery of a suite of internal standards vs. boiling point and water-air partition coefficient to account for matrix effects on the measured concentrations of VOCs in each sample. Though the method was originally designed around the vacuum distillation concentrator, the current study uses the method 8261 calculation model and different internal standard equilibration techniques to decrease measurement bias particularly of the heavier VOCs by purge and trap or static headspace analysis techniques.

P-35 Removal of Endocrine Disrupting Chemicals by a Constructed Wetland for On-Site domestic Wastewater Treatment

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Poster will discuss the development and implementation of an experimental system for treatment technology testing at the full-scale and track longer-term performance with respect to traditional regulatory variables such as, Nitrogen and Phosphorus speciation and dynamics. Pathogenic indicators will be evaluated. The tracking of longer-term performance for select EDCs, Steroid hormones, alkylphenol compounds with respect to traditional regulatory variables. Relate treatment efficacy to wetland performance characteristics.

P-36 The Permeability of Tarps and the Potential Influencing Factors

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Agricultural films are used during the fumigation of soils to enhance pest control and manage fumigant emission. The permeability of the films to fumigants varies widely with material type and manufacturing technique, as well as many other factors such as field conditions, temperature, fumigant type, film stretching, and moisture under the film. Permeability of twenty-seven (27) commonly-used agricultural films, including polyethylene films, "metalized" films, "virtually impermeable" films, and "totally impermeable" films from thirteen (13) manufacturers was determined, at 25oC and under ambient relative humidity, against methyl bromide, iodomethane, 1,3-dichloropropene, dimethyl disulfide, chloropicrin, sulfuryl fluoride, and methyl isothiocyanante. A database of the permeability of the tested films, expressed as Mass Transfer Coefficient (MTC), was established to provide a systematic way for growers to assess benefits of the films and for regulators to mitigate fumigation risk. Selected films were also tested at 40oC and at elevated relative humidity (at 25oC) to evaluate the effects of these parameters on permeability.

P-37 GPC Cleanup That Meets EPA Resolution Requirements without Chlorinated Solvents

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The current EPA Gel Permeation Chromatography Cleanup procedure (Method 3640A) requires 300mL of methylene chloride for each sample processed. Some alternate solvent systems are available for GPC cleanup, but strict column resolution requirements prohibit many laboratories from using non-chlorinated solvents for GPC cleanup. The EPA's current movement toward performance-based method criteria is removing many regulatory barriers to the development and use of new methodologies that are safer for laboratory personnel and for the environment while still producing accurate and repeatable results. This presentation shows that alternate column configurations with non-chlorinated solvents can still meet EPA requirements for resolution while handling high-lipid samples comparably to the traditional methylene chloride column.

P-38 GC-MS Identification Endosulfan Isomers and Its Sulfate Metabolite

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Endosulfan 3 EC was sprayed on field-grown pepper, melon, and sweet potato plants at the recommended rate. Plant tissue samples (leaves, fruits, and edible roots) were collected at 1 h to 30 days following spraying and analyzed for endosulfan isomers (α- and β-isomers). GC-MSD results indicated the formation of the molecular ions of m/z 170, 272, and 387 that correspond to endosulfan sulfate as the major metabolite of endosulfan and the relatively higher persistence of the β-isomers as compared to the α-isomer.

The initial total residues (α- and β-isomers plus endosulfan sulfate) were higher on leaves than on fruits. On pepper fruits, the α-isomer, which is the more toxic to mammals, dissipated faster (T1/2 = 0.9 d) than the less toxic β-isomer (T1/2 = 2.5 d).

P-39 Residue and Dissipation Pattern of Flonicamid and its Metabolites in Pepper During The Cultivation

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Residue and dissipation pattern of flonicamid (N-cyanomethyl-4-tripfloromethylnicotinamide) and its metabolites (TFNG and TFNA) was investigated in pepper during cultivation. The samples were collected twice a day after three times application of pesticide. The pesticide residues were extracted from samples by acetonitrile:water (1:1) solvent, liquid/liquid partitioned with ethyl acetate, and analyzed using UPLC-MS/MS. The percentage of recoveries varied from 84% to 93% in the concentration of 0.1 and 0.5 mg/kg of spiked samples. The residue levels were 0.75 and 0.02 mg/kg at 2 day and 30 days after the last application. During one-month period, the residue level of flonicamid in pepper decreased continually with increase in residue levels of the metabolites (TFNG and TFNA). A first-order model of the data revealed a fast initial dissipation (half-life 3.1 day). These results may be useful for understanding the dissipation patterns of flonicamid and its metabolites in agricultural products.

P-40 Characteristics of Deposit Rate for Different Formulations and Dissipation Pattern for 13 Pesticides in Peach during Cultivation

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Pre-harvest field experiments were conducted to characterize dissipation pattern of 13 pesticides consisted of different formulation and chemical property in peach. The pesticides were sprayed onto the peach and quantified after sampling at 0(2H), 1, 2, 3, 5, 7, 10, 15 and 21th day of different intervals. The residues of pesticides were analyzed with GC-ECD/NPD, LC-UVD and LC/MS/MS. Weight of peach during cultivation was increased approximately 70%. Deposit rate of sprayed pesticides on peach were appeared 0.3~1.1% in emulsifiable concentration(EC), 0.4~1.1% wettable powder(WP). Order of deposit rate was observed water dispersible granule(WG) > suspension concentrate(SC) > emulsifiable concentration(EC) > wettable powder(WP). Half-lives of 8 pesticides(azoxystrobin 6.7, cyazofamid 5.2, cyhalothrin 8.0, cypermethrin 7.7, diflubenzuron 7.9, etoxazole 5.0, imidacloprid 6.5 and thiacloprid 5.9 days) were showed 5 to 8 days. Iprodione and thiamethoxam were dissipated rapidly and showed 3.3 and 4.4 days half-lives respectively. But some pesticides showed longer than 10 days, pyraclostrobin 10.4, acetamiprid 10.6, buprofezine 12.2 and boscalid 12.3 days. Half-lives were observed low correlation to Log P characteristics of each pesticides. Half-lives were affected by increasing rate of crop weight.

P-41 Use Patterns of Neonicotinoid Insecticides on Cucurbit Crops and their Potential Exposure to Honey Bees

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Residues of the neonicotinoid insecticides: imidacloprid, dinotefuran and thiamethoxam and their metabolites as well as the *N*-methyl carbamate oxamyl were determined in foliage, pollen and nectar collected from pumpkin (*Cucurbita pepo* L.) fields treated at label-approved application rates using different application methods. Detected residues of applied neonicotinoids varied depending upon the type and timing of application. The highest residues were found in pumpkin foliage, followed by pollen, then nectar. Residues were highest for foliar and chemigation applications applied when flowers were present. The *N*-methyl carbamate oxamyl was found on pumpkin foliage and was not detected in pollen and nectar. The experiment was conducted over two growing seasons to assess potential exposure to neonicotinoids, a class of pesticides that may cause disorientation and associative learning problems in honey bees.

P-42 Testing for Aflatoxins in corn and peanut butter using QuEChERS strategy

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Many reference methods are available for analysis of aflatoxins. Most popular methodologies for routine analysis include the use of immunoaffinity columns for sample cleanup and HPLC with fluorescence detection for analysis. In an attempt to avoid using immunoaffinity columns, we applied the QuEChERS methodology to the sample preparation for analysis of aflatoxins B1, B2, G1 G2 in peanut butter and corn. QuEChERS stands for Quick, Easy, Cheap, Effective, Rugged, Safe and is used as a dispersive SPE (dSPE).

It was developed and is widely used for cleanup of samples during analysis of pesticides in fruits and vegetables. We employed the new effective fat-removal sorbent in combination with another common SPE sorbent to achieve the desired cleanup results during analysis of aflatoxins. Corn and peanut butter samples were extracted using 84% acetonitrile –16% water solvent. Then the extracts were spiked with aflatoxins and cleaned using dSPE and analyzed by HPLC-FL using TFA pre-column or bromine post-column (Cobra cell) derivatizations. The results indicated that excellent recoveries (above 88%) and reproducibility (below 13% for G1 and below 4% for other compounds) can be obtained by using a new method.

P-43 The Use of UPLC-MS/MS for the Detection of Challenging Veterinary Drugs and Growth Hormone Promotors

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Several classes of chemical compounds are classified under the generic term of "veterinary drugs" and this can be challenging when developing analytical methods. It has also been recognized by regulatory bodies, that there are a lack of appropriate quantitative efficient methods. Here UPLC-MS/MS is investigated for the analysis of some of the more challenging veterinary drugs.This poster addresses some of the challenges associated to the compounds: 1) dexamethasone & betamethasone which are synthetic glucocorticoids used in animal husbandry and also 2) recombinant bovine somatotropin which is a growth hormone used in some countries as a general growth promoter in pigs and cattle but also in lactating cows to increase milk production. These challenges include maintaining chromatographic separation of the epimers in a short time-frame, testing for the presence of protein-based compounds in animal samples, and the detection of both examples at low levels in complex matrices such as liver.

P-44 Analysis of Paralytic Shellfish Toxins in Bivalve Mollusks using HPLC Method with Post-column Derivatization and Fluorescence Detection

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Mouse Bioassay to detect dinoflagelate derived neurotoxins has major drawbacks that led to exploration of chemical methods of analysis. Recently, HPLC method that utilizes post-column oxidation of toxins under alkaline conditions has been approved as a new official AOAC method – OMA 2011.02. Three groups of toxins are separated on C18 column using a single step-gradient method. Products of post-column derivatization of toxins can be detected with high sensitivity using a fluorescence detector leading to determination of toxins type and concentration. We describe the use of Pickering Laboratories post-column derivatization system for analysis of paralytic shellfish toxins according to AOAC Method 2011.02

P-45 Screening for Pharmaceuticals and Personal Care Products in Drinking Water Using a 3-D Iontrap and Automated Online Sample Preparation

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Pharmaceuticals and personal care products (PPCP) consist of human and veterinary drugs, consumer products such as sun-screens, nutritional products, and cleaning products. These compounds enter the environment through a variety of sources including wastewater effluent, landfill leachate, industrial effluent, and animal feed lots. Environmental impact of these compounds remains unclear. However, in 2007 the EPA released method 1694 addressing the analysis of these compounds. This method utilizes solid phase extraction (SPE) followed by LCMSMS monitoring a single transition. We will demonstrate automated online sample preparation and automated data processing using 3-D iontrap full scan data for quantitation and data dependent (DD) full scan MS2 spectra for confirmation of PPCPs. 1mL injections of standards in HPLC grade water were analyzed with no additional preparation, PPCPs were detected down to levels as low as 10 ppt in standards, several sources of drinking water were analyzed, and caffeine was quantitated in bottled beverages.

P-46 Automated Solid Phase Extraction of Pharmaceuticals and Personal Care Products in Surface and Waste Water Samples - Method Development and Validation

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Aquatic samples require the analysis of pharmaceuticals and personal care products (PPCPs) target compounds are usually prepared by solid phase extraction (SPE) methods using different adsorbents. The sample preparation will usually take about two days to complete and would have subjected to many tedious steps that are prone to mistakes. The ability to automate the SPE process would minimize steps required in the SPE sample preparation and improved the data quality and operation efficiency. We present in this talk results obtained during the development and validation of an automated SPE sample preparation method that has been used to prepare aquatic samples for liquid chromatography/tandem mass spectrometric analysis of PPCPs. Used in the monitoring program of the 2010 PPCP typical background study in Ho Chi Minh City (HCMC), Vietnam, a total of 62 PPCP target compounds were monitored using this automated SPE with quality control data in two different laboratories. Quality assurance data obtained during the analysis will be discussed on the ruggedness, including precision and accuracy, of this automated process.

P-47 Novel Atmospheric Ionization Techniques, APGC and ASAP, used in the Analysis of Pesticides and Fungicides

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Atmospheric pressure ionization has primarily been used to interface MS with LC, but it is also a powerful ionization method that can be applied to GC and probe analysis. Novel atmospheric pressure ion sources, Atmospheric Pressure GC (APGC) and Atmospheric pressure Solids Analysis Probe (ASAP) are briefly described. ASAP results on a fungicide, Azoxystrobin, used in wheat farming are discussed. Approximately 100 pesticides, including organochlorine, organophosphorus, and organonitrogenated compounds were used to evaluate the APGC source. Two ionization mechanisms of APGC will be described. Results show a wide-scope screening method can be employed by using the proton transfer technique. Applying this technique to pesticide screening in foods (nectarine, orange, spinach) allowed the presence chlorpyriphos ethyl, deltamethrin, and endosulphan sulphate to be confirmed. In combination with high resolution TOF MS, APGC provides highly sensitive and selective detection of a wide range of analytes in food and environmental samples.

P-48 Optimization of the quantity of octadecyl (C18) and Primary Secondary Amine (PSA) using a QuEChERS -type extraction for the development of a multi-residue/multi-class method to determine pesticide residues in milk using experimental design

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A fast and selective multiresidue/multiclass method for the determination of pesticide residues in milk was developed using gas chromatography with mass spectrometric detection and with a programmed temperature vaporization solvent vent injector. Sample preparation procedure was based on the QuEChERS method and was optimized by applying experimental design for the selection of the sorbent (octadecyl and primary – secondary amine). For the determination of precision (RSD%) and accuracy (as a recovery percentage), guidelines given by the SANCO/10684/2009 document were followed, taking five samples at the MRL of the pesticide and five samples at five times the MRL. An analytical curve was made with five concentration levels (n=4), obtaining a linear range between 1 - 100 µg/L. LOQs and matrix effects were also assessed. Generally, the resulting data matched regulatory requirements, showing recoveries between 70 - 120% and an RSD<20% for the majority of pesticides studied. Therefore, the optimized method for milk shows a good performance in terms of linearity and precision. Finally, the validated method was successfully applied to real samples from milk farms of the department of Antioquia (Colombia).

P-49 Analysis of current use pesticides in human milk at part per trillion levels using GC/MS-NCI and LC/MS/MS-ESI techniques

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The analyses of human milk pose unique challenges in addressing limited sample size, variation in lipid content, and background contamination. These among other issues not normally encountered when analyzing foods resulted in the reconsideration of the use of matrix matched standards and the need for low detection levels. A multi-analyte method based on QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) is being developed to analyze current-use pesticides along with selected common household contaminants in milk. A single five gram milk sample is extracted and analyzed using low resolution mass spectrometry together with selective ion monitoring and matrix matched standards employing combinations of LC/MS and GC/MS techniques from NCI (negative chemical ionization) and MS/MS to achieve detection levels at the parts per trillion ranges. This method results in approximately 90% extraction efficiencies and achieves low detection levels with minimal to no matrix interferences.

P-50 The Quechers Extraction Approach And Comprehensive Two - Dimensional Gas Chromatography Of Halogenated Persistent Organic Pollutants In Cow Milk And Human Breast Milk

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Persistent organic pollutants (POPs) are a group of chemicals that include halogenated pesticides, brominated diphenyl ethers (BDEs) and polychlorinated biphenyls (PCBs). Due to the lipophilic nature of these components they accumulate in the fatty tissue of animals and bioaccumulate up the food chain. According to the World Health Organization human breast milk is an ideal matrix to monitor the levels of POPs in not only the mother and infant, but also as a key indicator of the levels of these chemicals in the local environment. Current methodology for the analysis of halogenated pesticides, PCBs and BDEs can be expensive, solvent intense and time consuming. The QuEChERS extraction approach coupled to a silica cartridge SPE cleanup may be an attractive sample preparation alternative for biomonitoring efforts for halogenated POPs in milk. Comprehensive two-dimensional gas chromatography (GCxGC) with an electron capture detector (ECD) may also offer a more cost-effective alternative. Method development was done using whole cow milk and later compared to a NIST Standard Reference Material of Human Breast Milk.

P-51 LOD "Limbo" - How Low Can You Go?

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The achievement of lower limits of detection (LOD) for pesticide residues in fruits and vegetables has always been a primary goal for residue laboratories. With the previous instrumentation in our laboratory, LODs were good but not great. With the addition of two, GC Triple Quadrupole instruments, our lab has significantly reduced the LODs for all of the compounds we were previously analyzing. We have also been able to increase the number of pesticides in our screen due to the triple quadrupole instrument reducing and, in some cases, completely eliminating the various matrix effects on most of those compounds. This experiment will compare LODs between GC Triple Quad and other GC Mass Spectrometry techniques.

P-52 Quechers Approach to Honey Analysis using Triple Quadrupole GCMS

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A rapid and inexpensive multiresidue method for pesticides in honey is presented. Extraction of a 15g sample, utilizing a QuECHERS APPROACH was used. Extracts were concentrated to a 2 g/mL. Analyses were previously conducted by GC/MS, GC/XSD and LC/QqQ/MS. Now the Method has been further stream lined to utilize only our Triple Quadrapole instruments both GC and LC. The single quadrapole GCMS and GC/ XSD were replaced by a Triple Quadrapole GCMS. This change also lead to 11 new compounds added and 5 other compounds being removed from the screening list. LODs for the GC compounds were significantly lowered to 10 ppb for most compounds, and the LOQ's were also lowered significantly overall..While using this new method multiple pesticides have been determined to be present in honey samples collected for analysis.

P-53 Optimized method for analysis of emerging contaminants from water samples using SPE and LC-MS/MS

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Nowadays, there is a great concern about environmental contamination by emerging contaminants due the potential risk to human health and to aquatic ecosystems. The principal contamination sources of these compounds are agricultural, industrial and domestic activities, with the courses of surface water and groundwater inevitably being the principal destination. To have an appropriate evaluation of the ecological risks and to guarantee the quality of drinking water, in this work, methods to determine some emerging contaminants, namely some pharmaceuticals and pesticides, were developed. Solid phase extraction procedures were evaluated by the use of different types of solid phases (commercial C18 and Oasis HLB; lab-made fluorinated and phenyl) with subsequent analysis by LC-MS/MS. Separate extraction and analysis methods were developed for the pharmaceuticals and the pesticides, and both resulted in satisfactory recovery values and quantification limits in the range of ng/L. FAPESP, CNPq, INCTAA

P-54 Detection and Identification of Unknown Contaminants during ToF Screening for Pesticides in River Water using an Integrated Software Approach

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When analysing environmental waters, pesticide contamination screening is one of the most important analyses carried out. However, other contaminant species, such as veterinary drugs or human pharmaceuticals and their metabolites, may also be present at similar ultra-trace levels as pesticides and could be equally as harmful to the aquatic ecosystem. Discovery of a non-

targeted compound subsequently entails the confirmation and identification of that compound. Accurate and precise exact mass data on both the low energy precursor ion and the MSE high energy fragment ions, together with an integrated software approach, provide increased confidence in the identification of the non-targeted species. This talk describes the non-targeted screening of water samples using OASIS HLB cartridges for SPE clean-up and pre-concentration, followed by analysis using Waters ACQUITY UPLC coupled with Xevo G2 QTof. Three non-targeted contaminant species were detected and successfully identified in river water using ToF screening and a structural elucidation workflow.

P-55 Pesticide monitoring to meet regulatory requirements by the direct injection of drinking water

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In recent times the use of pesticides, herbicides and fungicides has steadily increased, in an effort to meet global food demands. A percentage of such chemicals applied to crops will, inevitably, end up leaching into the surrounding soil and waterways. Strict environmental monitoring is mandated at a local or federal level, which endeavors to sustain or improve water quality, preserve the environment and protect human health. Tandem quadrupole mass spectrometers provide the 'gold standard' for ultra trace analysis, offering a combination of high selectivity, high sensitivity and the greatest confidence in results. The performance of some of these instruments has enabled some methods to dispose of sample preparation, improving laboratory productivity, with drinking water samples being stabilized and then directly injected onto the LC/MS/ MS instrument. However, difficulties remain when trying to discriminate against matrix components that exhibit similar physiochemical properties. Unawareness of these difficulties in each sample can lead to poor quality results and can impact on a laboratory's performance and reputation. Conventional tandem quadrupole systems when operated in multiple reaction monitoring (MRM) mode do not allow the direct monitoring of the sample matrix during routine high sensitivity determinations. A novel high performance tandem quadrupole instrument coupled with ACQUITY UPLC, along with TargetLynx software data processing, was used to rapidly determine pesticides with the direct injection of 100µL drinking water. All MS parameters were recalled from Quanpedia – a "one stop" database for storing and sharing user-defined acquisition and associated processing methods for the quantification of named compounds. A generic UPLC acidified water/ methanol gradient was used, with a total run time of 10 minutes. A unique capability of monitoring the matrix simultaneously, without compromising the performance, during ultra trace multi-residue pesticide analysis was also investigated. Pesticides were successfully determined with high precision at the ng/L level with the ability to detect to pg/L concentrations.

P-56 UPLC-APPI-MS/MS for High Sensitivity and High Throughput Analysis of US EPA Sixteen Priority Pollutants Polynuclear Aromatic Hydrocarbons

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Many PAHs are carcinogens and are closely monitored in the environment. The USEPA have formulated regulations for the monitoring and control of PAHs and have developed methods for their measurement in air, water, food, and other matrices. The regulated analytical methods include HPLC-UV/FLD (EPA Method 550, 8310), GC/FID (Method 610, 8100) and GC/MS (Methods 525, 625, 8270, 8275). In this work, we demonstrate UPLC-APPI-MS/MS for high sensitivity and high throughput analysis of USEPA sixteen priority pollutants PAHs. This method overcomes the problems of thermal decomposition and adsorption to GC inlet for analysis of low volatility PAHs by GC/FID and GC/MS. It also improves sensitivity, selectivity and offers much higher sample throughput relative to traditional GC/FID, HPLC-UV/FLD and GC/MS methods. An Acquity UPLC-TQD equipped with APPI source was used. The parameters such as MRM transitions, cone voltage and collision energy were automatically optimized with PAH tuning standard mix solutions by a flow injection using the IntelliStart program of Acquity UPLC Console. These tuning standards, prepared in pure chlorobenzene, contained PAH analytes with distinctive molecular weights (MW). Chromatographic separations were achieved on a Zorbox Eclipse PAH, 2.1 x 50 mm, 1.8 µm particle size, UPLC column, by gradient elution using acetonitrile/water as a binary mobile phase. The mobile phase solvents were 90:10 water/acetonitrile (A1) and 100% acetonitrile (B1). The mobile phase flow rate was 650 µL/min. The dopant chlorobenzene flow rate was 65 µL/min. In comparison with the existing US EPA methods, this method improves instrument sample throughput by at least 10 folds.

P-57 Determination of Banned Dyes in Food Sources by QuEChERS Methodology and LC/MS/MS Analysis

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International agency for research on cancer classifies the Sudan dyes as class 3 carcinogens and therefore it is illegal to use them as food additives, according to the FDA and EU. Laboratories performing analysis of Sudan dyes are not required to follow defined methods. However the EU has set detection limits for these dyes at 0.5-1 ppm.

The QuEChERS methodology and a superficially porous LC column were employed to extract a series of dyes from food sources well below the established limits of 0.5 ppm. The QuEChERS procedure offers a "just enough" sample preparation technique for the extraction of illegal dyes from difficult sample matrices like spices. The use of a superficially porous LC column is extremely well

suited for use with more complex samples, with efficient mass transfer. The combination of QuEChERS and superficially porous column equates to faster analysis time, higher throughput with optimum results.

P-58 Screening Method for 30 Pesticides in Green Tea Extract Using Automated Online Sample Preparation with LC-MS/MS

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As global agricultural trade grows, food safety and wellness concerns are at an all-time high. Currently, it is routine to analyze additives and contaminants, such as pesticide residues, in food products. For pesticide analysis, a common approach is to develop a fast multi-residue method which is capable of identifying and quantifying a large number of analytes in various food matrices. In this study, we describe an easy, comprehensive, online screening LC method using a Transcend TLX-1 system powered by TurboFlow technology to analyze multiple pesticide residues in green tea extract. The lower quantitation limits for all analytes are well below the maximum residue limit (MRL) set by the European Union (EU) and the Japanese government. Matrix effects and carryover are also discussed.

P-59 A Turn-key Solution for Automated Detection of Organic Contaminants in Food Matrices and Economic Adulteration

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The study of organic contaminants in food matrices includes target screening (a) Known Knowns, and untarget screening (b) Known Unknowns and (c) Unknown Unknowns compounds. Screening of food to protect the safety of public is of great importance. A new Food Safety Modernization Act (FSMA) law was signed by President Obama in January 2011 for this purpose. Accurate mass measurements of unknown compounds were determined in different matrices at various concentrations vs. control samples. SIEVE software was using for Experiment vs. Control analysis, the high ratio masses (Experiment/Control) were searching by Chemspider databases for contaminants in food and economic adulteration.

P-60 Analysis of 400+ Pesticides in a Single Run Using Triple Quadrupole Mass Spectrometer

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Increasing food safety concerns and the growing agricultural trade has resulted in stringent pesticide regulations globally. To comply with strict food safety standards, a screening and quantitative method for large numbers of pesticides is becoming important. This poster describes a method for analysis of multi-class pesticides in food samples using liquid chromatography coupled with triple quadrupole mass spectrometer. A multi-residue method was developed for screening and quantitation of approximately 400 pesticides in one 45 minute run. One or two ion ratios were used to confirm each analyte. In addition, the Quantitation Enhanced Data-Dependent (QED) scan that delivers an information rich mass spectrum while undergoing quantification could also be used for structural confirmation. The method was applied to pesticide analysis in orange, asparagus and apple extract. Furthermore, the method was developed using software with built-in workflows for streamlining method development and routine analysis. The experiment results will be discussed in detail.

P-61 A rapid solution using EQuan Orbitrap technology for combined qualitative and quantitative analysis of known and unknown pesticides in Water

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Exactive mass analyzer in combination with online solid phase extraction and novel software application was used to detect polar and nonpolar compounds with unified qualitative and quantitative data processing. EQuan MAX(tm) online solid phase extraction system was coupled to Exactive(tm) benchtop Orbitrap. Samples of surface water were spiked with a mixture of pesticides. From each of these samples 1 mL was loaded directly and without any treatment onto the stack of trapping columns. Detection was carried out in full scan mode alternating positive and negative ionization. Twenty pesticides representing a broad range of polarities were taken for analysis. The coupling of two trapping columns of different polarity yielded in highly sensitive detection of all analytes with excellent precision and reproducibility. All analytes yielded good linear behavior over the full range of calibration and showed good reproducibility in repeated injections. Automated untargeted and unknown screening was performed.

P-62 Quantitation of Organophosphate Insecticides in Drinking Water Using Automated Online Sample Preparation and a 3-D Ion Trap

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Organophosphates are irreversible acetylcholinesterase inhibitors and as such are highly toxic. Due to both the toxicity and other

effects such as; delayed learning rates in children, an increased risk of Alzheimer's, and chronic fatigue symptoms it is essential to be able to detect even low levels of these compounds. This presentation will demonstrate the use of automated online sample preparation coupled with a 3-D iontrap mass spectrometer, performing full scan MS2, for the detection of organophosphates in drinking water. Calibration curves were generated from 1mL or 4mL injections of standards spike into HPLC grade water with no further preparation. Quantitation was based on the post acquisition extraction of multiple fragments, where possible, for each compound from the full scan MS2 data. Several local water sources were also analyzed.

P-63 Utilization of High Resolution LC-MS for Screening and Quantitative Analysis of Pesticides in Food Matrix using a Q-Exactive Bench Top Orbitrap Platform

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Current methodologies for quantitation of pesticides in food revolve around using triple quadrupole platforms. The method described here utilizes LC-MS/MS with QExactive using Selective High Resolution Accurate Mass. The QExactive is capable of resolutions of up to 140,000K FWHM. A Thermo Hypersil Gold aQ 50x2.1mm 1.9u column was utilized with a run time 12mins. A standard curve spiked with 110 pesticides in neat matrix ranging from 0.05 to 250 ng/mL was injected in triplicate. Analysis of hot peppers and bell peppers was screened for targeted and unknown pesticides. A full scan was set to 70,000K resolution to minimize matrix interference while Data Dependant MS/MS was collected and compared to spectra library for confirmation and help prevent false positives. Calibration lines show r2 better than 0.99, limits of detection varies from 50 to 250pg/mL depending individual compounds. The poster will show online searching and quantitation capabilities with one streamline software.

P-64 Rapid Analysis of 303 Pesticide Residues in Green Beans Using Triple Quadruple GC-MS/MS

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Both GC with its element selective detectors and single quadruple GC/MS are the common techniques used for GC-amenable pesticide residue analysis in most labs. However, as the number of pesticides increase and the MRLs are lowered, the complete analysis of one sample has to be carried out by several injections. Different methods are employed on multiple instruments to analyze hundreds of pesticides, then are confirmed using different types of GC columns for the pesticide residues found in the samples. To increase the sample throughput and accuracy in matrix, the Thermo Scientific TSQ Quantum GC triple quadruple was used in this experiment, because it has the ability to acquire 3000 timed-SRMs per analytic run. This method uses one injection to screen and confirm hundreds of pesticides with at least two SRM transitions for each compound according to the European Council Directive 96/23 /EC4. The combination of the triple quadrupole GC/MS/MS and Ion Trap GC/MSn with QuEChERS sample preparation method has been investigated for pesticide analysis in different food matrixes. However, SPE as a clean-up procedure is still the official analytical method for pesticide residues analysis in some countries. This poster demonstrates the method for analysis of 303 pesticides in green beans by using acetonitrile extraction, followed by the SPE clean-up step and finally determination by the TSQ Quantum GC-MS/MS in the timed-SRM mode. The linearity, average recoveries and precision of all 303 pesticides at 0.010 mg/kg in green beans have been evaluated.

P-65 Determination of Organochlorine Pesticides in Carrots by QuEChERS and GC-ECD

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The organochlorine pesticides (OCPs) due to their extensive use and also because of their chemical stability and slow biodegradation became ubiquitous pollutants. Most of the OCPs are therefore included in the persistent pollutants which are characterized by long half-lifes in the environment and to have potential biomagnification capacity through food chain. The aim of this study was to develop a method to evaluate OCPs in carrots. The analyzed OCPs include: HCH(α,β,δ), HCB, lindane, aldrin, 4.4'-DCBP, α-endosulfan, dieldrin, p-p'-DDE, endrin, β-endosulfan p-p'-DDD, o-p'-DDT and methoxychlor. The extraction was performed with QuEChERS. GC-ECD was employed for the determination of pesticides. With the chosen QuEChERS linear calibration curves were obtained for the 14 OCPs. Concentrations ranging from 5 to 70µg/kg in carrots were studied. The samples homogenization influences the results reproducibility therefore it can be a crucial step for the analysis. The sample preparation, QuEChERS, can be applied to carrots for OCPs analysis.

P-66 Development of Analytical Method for Determination of Pesticides in Soils by UFLC/MS

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Traditional pesticide analysis in soil are known to be laborious and time-consuming methods. The aim of this work was to development an easy, rapid and low-cost sample preparation approach for the determination of 24 pesticides in soil by ultra fast liquid chromatography-electrospray-mass spectrometry. For this purpose, three solvents (methanol, acetonitrile and ethyl acetate) and three buffers (acetate, citrate and carbonate) were compared based on ultrasound-assisted extraction methods. The purpose of this metodology consider acetonitrile ultrasound-assisted extraction using acetate buffer and centrifugation. This method does not account any clean up. The recovery studies were performed over different soil blanks spiked at three concentration levels. The best recoveries were found ranging from 75% to 110%. In this study, all pesticides have shown a relative standard deviation below 20%. Finally we test the method using four soils of different physicochemical properties and no statistical differences ($p>0.05$) were found between the recoveries obtained.

P-67 Comparison of Sample Preparation Methods Involving Different Versions of Quechers for Determination of Pesticide Residues in Strawberries by LC-MS/MS

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Multiresidue pesticide analysis in foods has acquired great importance in the development of analytical methods due to environmental concerns and consumer health. The strawberry crop has a great economic importance due to its functional properties but is one of the most contaminated foods in Brazil. The objective of this work is to evaluate and compare the sample preparation methods for 29 residues of pesticide selected for their frequent use in Brazil using different versions of QuEChERS, Original (2003), AOAC (2007.01), CEN Standard Method EN 15662 (2008) and a modified version of the EN 15662 in strawberries, with determination by LC-MS/MS. Preliminary results show that the modified version of the EN 15662 method showed better values of average recoveries for most pesticides, in the 70–120% range, with RSDs ≤20%, as recommended for analysis of pesticide residues, and better clean-up of samples. CAPES, CNPq, FAPESP and INCTAA.

P-68 Determination of Pesticides in Water by Solid Phase Extraction (SPE) and UFLC/MS Using a Char of Vegetable Origin as the Stationary Phase

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In the last decade have been widely used stationary phases based on carbon for extraction and concentration of pesticide residues in water. In this study the performance of a char of vegetable origin (CVO) is compared with commercial graphitized black carbon (GBC). The performance was evaluated through recovery studies using SPE methodology. CVO was obtained subjecting plant material at 873K (3h), the carbon material obtained was blended with diatomaceous earth (70:30) and this blend was used as stationary phase. The GBC also was mixed with earth diatomaceous. The SPE was performed by spiking piped water samples (pH 5.3) and by using methanol and acetonitrile as eluents. The results showed that recoveries of CVO were between 70.2% and 105.0% for target pesticides, with %RSD between 6.0% and 15.3%. Recoveries obtained with this carbon were greater than those obtained using the commercial GBC (recoveries between 59.5% and 97.5%).

P-69 Evaluation of Different QuEChERS Approaches for the Analysis of Pesticides in Paddy Rice by LC/MS/MS

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There are different analytical methodologies for pesticide residue analysis in paddy rice, as pressurized-liquid extraction, gel permeation chromatography and supercritical-fluid extraction. Previously, we compared different QuEChERS approaches for the determination of 42 pesticides in polished rice. The suitability of these QuEChERS methodologies for the analysis of 47 (17 insecticides, 13 fungicides and 17 herbicides) pesticide residues in paddy rice, a complex and different matrix than polished rice, are compared in this communication. The performance of each method (linearity, limits of detection and quantification, recoveries, repeatability and matrix effects) was evaluated at 20 and 200 ug/kg by LC-QqQ/MS. The original and citrate buffered versions of QuEChERS gave the best results for 81 and 85% of the pesticides assayed respectively. In specific cases, depending on the analyte, one method is preferable than the other. For example, diazinon was best determined through original QuEChERS but the method of choice for fipronil was citrate QuEChERS.

P-70 A Thermodynamic - Extended UNIQUAC Model for Salting-out Assisted Liquid-Liquid Extraction

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QuEChERS method sparked a revolution in the area of sample preparation for the analysis of pesticide residues in food. The backbone of the QuEChERS method, dispersive SPE clean up, has been adopted by other methods. As with the QuEChERS method, most of these methods use salting-out assisted liquid-liquid extraction (SALLE). NaCl- MgSO4 is well accepted. However, the ratio of two salts, and the ratio of liquid-salt differs among methods. A thermodynamic model is necessary to explain the mechanism of SALLE, and to allow for method optimization, modification and troubleshooting. Based upon IRAM method (Irvine Rapid Analytical Method), this study uses extended UNIQUAC model to explain the dissolution of NaCl-MgSO4 and re-crystallization of Na+, Mg2+, Cl - and SO4 in aqueous phase. Through a quaternary phase diagram, re-crystallization products can be found. With the product of re-crystallization, water and organic solvent, a ternary phase diagram was worked out. An optimized salt-water-acetone ratio was calculated. This optimization is demonstrated by the experimental data generated to support the IRAM method which is soon to be submitted in review as an LIB.

P-71 Evaluating Porous Materials for Sampling Pesticides from Surfaces using DART-MS

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Rapid screening of pesticides present on the surface of leaves, metal and building materials has been facilitated by using a direct analysis in real time (DART) open air surface desorption equipped mass spectrometer. These experiments focus on the use of various materials to collect pesticides from larger objects including plants and machinery by using a vacuum assisted sampling approach. Evaluation of the efficiency of various polymeric foams, plastic screens and wire screens for capture of analyte with and without the use of solvents will also be examined. Suitability of different materials as both sampling and desorption ionization support will be reported. These experiments follow on original pesticide screening experiments where polyethylene foam was used as both the collection and desorption substrate present on the surface of small fruits and nuts as completed using "Transmission-mode" DART-MS analysis[1]

1. Edison, S.,, et al., Rapid Commun. Mass Spectrom., 2011, 25, 127-139

P-72 Screening and Identification of Non-targeted Compounds with Confidence Using an Exact Mass Spectral LC-MS/MS Library

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Pesticide and other xenobiotic screening of non-targeted compounds is increasingly being performed by full spectrum accurate mass measurement mass spectrometers. These instruments are sensitive and, in LC/MS, provide pseudo molecular ion information often sufficient to determine a molecular formula. By searching a database with the either the measured mass and its isotopes or the molecular formula, compounds whose ions can easily be lost in the sea of matrix ions of a complex food sample, can be extracted from the data. However, algorithms to do so can be riddled with both false positive and false negative results. In addition, large databases will contain many isomers and the pseudo molecular ion information is not sufficient to determine the one present. MS/MS spectra at either multiple collision energies, or a set energy for screening, searched against an exact mass spectral library can reduce false positives and false negatives significantly. This presentation will describe a database with over 7500 compounds including pesticides, toxins and pharmaceuticals containing MS/MS spectra for over 2500 compounds collected at 3 collision energies. The effectiveness of searching LC/Q-TOF MS and MS/MS data for food samples extracted with QuEChERS the with this database and library will be discussed along with strategies for the experimental settings to collect the data.

P-73 Analysis of Fluoronated Alkyl Compounds in Bio-Solid Matrix by GC Q-TOF Mass Spectrometry

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Fluorotelomer alcohols are synthetic compounds used as building blocks for fluorinated polymers and could arise in the environment as intermediate degradation products from those same polymers. Furthermore, fluorotelomer alcohols oxidize to fluorinated carboxylic acids, some of which have been found to be toxic under typical environmental conditions. Sensitive and specific analytical methodology is required to study the fate and transport of fluorotelomers in the environment and gain understanding of human exposure and toxicity. Herein, we present a GC Q-TOF method for the analysis of fluorinated alkyl compounds in complex biosolid matrix. Analyte ions were easily extracted from the heavy matrix and comparison to MRM data collected previously suggests detection limits on the order of 5 – 500 fg on column.

P-74 Determination of Polychlorinated Biphenyl Congeners in Foodstuffs and Animal Feed by GC-MS/MS

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Polychlorinated biphenyls (PCBs) are ubiquitous, lipophilic organic contaminants that are globally present at low concentrations in foodstuffs and animal feed. Due to their toxicological potencies in humans and animals, rigorous performance criteria for their analytical determination are stipulated by legislation in the European Union and the United States. European legislation currently demands that the confirmation of dioxin-like PCB concentrations in food and feed have to be performed by gas chromatography coupled to high resolution mass spectrometry (GC-HRMS). In a study, the performance of a triple quadrupole GC-MS/MS system for the reliable, sensitive and unambiguous determination of dl-PCBs and non-dioxin like PCBs (ndl-PCBs) in food and feed was tested. Samples of meat, liver, milk, hen eggs and feed samples, previously analysed by GC-HRMS, were run on the GC-MS/MS system. The GC-MS/MS results demonstrate good linearity and response reproducibility over the range of interest as well as low detection levels both for dl- and ndl-PCBs. The results of this study also indicate that the Agilent GC-MS/MS system has the potential as an alternative confirmatory methodology for the determination of dl-PCBs in official food and feed control, pending analytical quality criteria to be set by legislative bodies.

P-75 Analysis of Trace Level Pesticides in Fruits and Vegetables by GC/MS/MS Using Agilent Ultra Inert Liners with Wool

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Trace level analysis of active and difficult pesticides in fruits and vegetables by GC/MS/MS was accomplished using Agilent Ultra Inert splitless liners with wool. With efficient deactivation on liner and glass wool, the Ultra Inert liners with wool provide excellent inertness resulting in good chromatography, superior sensitivity (1 ng/mL of LOQ in matrix) and linearity (over 1 – 100 ng/mL range with R2 > 0.99), excellent repeatability and performance stability for active compounds in different matrices. In addition, Ultra Inert deactivated wool increased productivity by keeping non-volatile matrix interferences in the liner, thus extending column lifetime and reducing frequency of MS source maintenance. The performance of Ultra Inert liners with wool was superior when compared to other liners used widely in pesticides analysis such as Restek Siltek Cyclosplitter liner. Ultra Inert liners with wool can be excellent choices for accurate analysis of pesticides in fruits and vegetables.

P-76 Analysis of Pesticide Residues in Rice Using QuEChERS AOAC Kit by LC-MS/MS Detection

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Rice consumption has increased exponentially worldwide, becoming the number one consumed food. Environmental samples from rice areas are well documented, but less information is known about pesticide residues in rice-grain samples. Traditionally, the determination of pesticide residues in rice has been accomplished by GC/MS. The application of new classes of pesticides has allowed laboratories to increase the use of LC tandem MS. This application note described the use of QuEChERS sample preparation approach for the extraction and cleanup of pesticide residues representing various pesticides classes in rice. The pesticides in the rice extracts were then determined by LC-ESI-MS/MS. The 5 ng/g limit of quantitation (LOQ) for pesticides in rice shown in this application was well below the maximum residue limits (MRLs). The mean recoveries ranged between 76% and 108% (average of 97.8%), with RSD below 10% (average of 4.7%).

P-77 High Mass Accuracy Provides Enhanced Selectivity for the GC/MS Analysis of Pesticides in Food

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The analytical power of GC/MS with high mass accuracy is useful in two broadly-defined ways: for the identification of unknown compounds, and to provide increased selectivity for the determination of target compounds, especially in complicated sample matrices. Whether the theoretical gains for the latter application can be achieved in practice depends greatly on the mass accuracy of the mass spectrometer, its mass axis stability over time, and the nature of the matrix interferences (relative retention time, mass, and magnitude.) We present some results obtained with typical food samples containing low levels of pesticides.

P-78 US EPA UPLC/MS/MS Method Development for Analytes of Concern

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The US EPA Chicago Regional Laboratory is developing methods for qualitative and quantitative determination for chemical weapon degradation products in soil and wipes, hormones, poisons and other analytes of interest at the ppb to sub-ppt concentration levels. These methods utilize pressurized fluid extraction, solid phase extraction and/or direct injection followed by UPLC/MS/MS analysis to achieve target quantitation levels. Proposed. preliminary and approved ASTM Standards will be presented to demonstrate the process and relevance of the work.

P-79 Proposed or Recent ASTM Standards for the Analysis of Alkylphenols and their Ethoxylates in Water by Liquid Chromatography/Mass Spectrometry

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Nonylphenol is an organic chemical produced in large quantities in the United States. It is soluble in water and moderately resistant to natural degradation in water. Due to its chemical properties and widespread use as a chemical intermediate, concerns have been raised over the risks it poses to both freshwater and saltwater organisms. Nonylphenol is now regulated in the effluent of sewage treatment plants (STP). States are beginning to include nonylphenol in their discharge permits. ASTM D7065 and D7485, which analyze for the short chain ethoxylates and nonylphenol, have been referenced for this analysis. Another Practice used by US EPA Region 5 Chicago Regional Laboratory (CRL) for monitoring the influent and effluent from STP was requested by a STP to make available through ASTM in order to screen for the influent or effluent from sources of alkylphenol polyethoxylates coming into the STP. There is interest in stopping the source of the longer chain alkylphenol ethoxylates from entering the STP in order to meet effluent guidelines. This Practice is a screening technique with qualified concentrations to determine the presence of longer chain ethoxylates in a water sample.

P-80 LC-MS Library System Protocol and Possible Future

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US EPA Region 5 Chicago Regional Laboratory (CRL) has been collaborating with the US EPA Office of Research and Development/ National Homeland Security Research Center, Office of Water/Water Security Division and partnered through a Cooperative Research and Development Agreement (CRADA) with the Waters Corporation, to build a searchable, transferable liquid chromatography-mass spectrometry (LC-MS) library that detects selected harmful organic compounds in drinking water. NIST is collaborating with EPA to build a LC/MS library searchable via the NIST softwate.

P-81 Rapid Screening of Grain Samples for Seed Treatment Pesticides Utilizing a Waters ASAP (Atmospheric Solids Analysis Probe) and Xevo Mass Spectrometer

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The Waters ASAP is reported to be a useful tool for the analysis of volatile and semi-volatile solid and liquid compounds under atmospheric pressure ionization (API) conditions. ASAP uses heated nitrogen to volatilize the sample from a glass capillary tip, which is then ionized with a corona discharge pin and an MS spectrum is generated without the need of sample preparation or chromatographic separation. The purpose was to evaluate the use of ASAP as a quick screening method for the analysis of a number of seed treatment pesticides. This analysis is routinely performed on an LC/MS/MS system. A Waters Xevo Mass Spectrometer was setup with an ASAP assembly and corona discharge pin operating in API. The method involved removing the probe from the ASAP assembly and loading the probe by either placing the glass capillary tip into the ground solid sample or applying a uL amount of liquid sample to the glass capillary. The probe is then inserted back into the MS and a spectrum acquired. The method was setup to screen for 20 seed treatment pesticides. The results were compared to those generated from the routine LC/MS/MS analysis with good agreement. This looks to be a quick and sensitive screening method of solid/ liquid samples which involves minimal sample preparation and no chromatographic separation.

P-82 Determination of 64 Organochlorine, Organonitrogen and Organophosphorus Pesticides in Olive Oil by Optimized QuEChERS Procedure

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This poster describes an optimized QuEChERS procedure for the determination of multi-class pesticide residues in olive oil. Sixty four pesticides belonging to organochlorine, organonitrgen and organophosphorus classes were included in this study. Olive oil samples were extracted, cleaned, concentrated and detected by GC/MS in SIM mode. Different oil/water ratios were compared and optimized to increase the recoveries of pesticides, especially for lipophilic compounds. Different amounts of acetic acid in acetonitrile were evaluated. 0.25% acetic acid in acetonitrile yielded comparable recoveries to 1% acetic acid and yet not harmed the GC column. Clean-up methods including dSPE, C18 SPE column, PSA/C18 dual layer SPE column and without clean-up were also investigated. Clean-up with PSA/C18 dual SPE column removed most co-extractives and thus protected GC system from contamination by non-volatiles extracted. Accuracy, precision and method detection limit studies were carried out under optimized condition.

P-83 Determination of Seven Problematic Pesticides in Olive Oil

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Seven problematic pesticides in olive oil, including six base sensitive compounds (chlorothalonil, dicofol, dichlofluanid, tolylfluanid, folpet and captan) and one acid sensitive compound (pymetrozine) were determined by modifed QuEChERS procedure and GC/MS in SIM mode. Different amounts of acetic acid in acetonitrile were evaluated. 0.5% acetic acid in acetonitrile resulted better recoveries than 1% acetic acid, even for those base sensitive compounds. Comparison of different clean-up methods including dSPE, C18 SPE column and PSA/C18 dual layer SPE column were also studied. Clean-up with PSA/C18 dual SPE column gave best clean-up effect. GC injector can be contaminated by the olive oil extract and need be cleaned when folpet and captan show decreased GC response. With optimized procedure acceptable recoveries (66-123%) and precisions (RSD <13%, n=4) were achieved for all seven problematic pesticides in olive oil.

P-84 Automated QuEChERS Extraction for the Determination of Pesticide Residues in Foods using Gas Chromatography/ Mass Spectrometry

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One of the most important aspects of reducing pesticide exposure is monitoring of pesticide residues in foods. A number of analytical methods have been developed, many of them based on traditional liquid-liquid extraction in combination with GC-MS or LC-MS. The QuEChERS sample preparation methods have been developed to help monitor pesticides in a range of food samples. These methods, however, still require many manual steps, such as shaking, mixing, centrifugation, and dispersive SPE (dSPE), making them quite labor-intensive. There is a need for automating the dSPE technique to clean up QuEChERS type extracts in order to improve laboratory productivity for monitoring pesticide residue in foods. This paper describes an automated dSPE cleanup method for QuEChERS extracts that is performed with commercially available kits from Agilent® Technologies. Extraction and clean-up is performed using a micro-scale version of this method and automation is achieved using a GERSTEL MPS autosampler equipped with an Anatune CF-100 centrifuge. The clean-up process is followed by automated injection of the cleaned extract to a GC/MS system. Analytical methodology for confirming the presence of a variety of pesticides in various food samples was developed using an Agilent 7890 GC with 5975 MSD. Samples were introduced into the gas chromatograph using a GERSTEL Thermal Desorption Unit (TDU) and MPS autosampler with Automated TDU-liner Exchange (ATEX) option. The sensitivity and selectivity of GC/MS combined with the described injection technique, results in method detection limits that meet acceptance criteria for reporting maximum residue levels (MRLs) as established by regulatory agencies. The ability to automate the dSPE clean-up of QuEChERS extracts and to couple extraction and clean-up directly to GC-MS analysis, results in improved laboratory productivity by streamlining the complete analytical process.

P-85 Screening and Quantitation of pesticide residues in food using LC-MS/MS and Identification using MS/MS Library Searching

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The traditional LC-MS/MS method setup to determine pesticide residues uses two MRM transitions for quantitation and the MRM ratio for identification, which has limitations due to matrix interferences. The screening method introduced in this presentation uses the retention time and MRM transition of each targeted pesticide, and full scan MS/MS spectra automatically collected during the screening. MS/MS spectra are searched against a library to reduce false positive results caused by matrix interferences. The developed method was used to screen QuEChERS extracts of fruit and vegetable samples for pesticide residues. A HPLC system with a small particle size reversed phase column was used for separation. A single optimized MRM transition was monitored for each pesticide for quantitation and automatically acquired MS/MS spectra were used for compound identification. The samples were analyzed on different hybrid LIT – QQQ and QQQ – TOF mass spectrometers to verify the library and to investigate the possibility of using it on different mass spectrometers.

P-86 Non-Targeted Screening for Food Residues using High Resolution Accurate Mass LC-MS/MS and Statistical Data Processing

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Recent regulations on food analysis require the screening for pesticides using confirmatory techniques, such as GC/MS and LC-MS/MS. With more than 1000 pesticides there is a demand for powerful and rapid analytical methods, which can detect very low concentrations of pesticides in a variety of food matrices. Here we present a high-throughput LC-MS/MS method that combines multi-class compound screening with identification based on accurate mass MS/MS library searching. Furthermore, acquired full

scan MS data was used to retrospectively screen for non-targeted and unexpected contaminants. Fruit and vegetable samples were extracted using a QuEChERS procedure and injected into LC-MS/MS after dilution to minimize possible matrix effects. LC separation was performed using a Shimadzu UFLCXR system with a Restek Ultra Aqueous column. An AB SCIEX TripleTOF™ 5600 system was used to detect TOF-MS and MS/MS.

P-87 Determination of Pesticide Residues in Vegetable Juices using the Triple Quadrupole LC-MS/MS and GC-MS/MS, and a Single Quadrupole Mass Spectrometer GC-MS

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The study evaluated the capabilities of mass spectrometry (MS) in combination with gas chromatography (GC) and liquid chromatography (LC) for the determination of a multitude of pesticides. GC-MS/MS and LC-MS/MS multiple reaction monitoring (MRM) methods and a GC-MS selected ion monitoring (SIM) analytical method have been established for determination of 39 pesticides in vegetables and fruits juice blends. The samples were prepared using QuEChERS AOAC method. The extracts were analyzed on the GC/QQQ, LC/QQQ and GC/Q and results were compared. The assessment showed a wider scope and better sensitivity when detection is based on LC /QQQ and GC/QQQ. The GC/Q was also operated in scan mode. Scan results were evaluated using Agilent's Deconvolution Reporting Software (DRS). Samples obtained through the Grocery Manufacturers Association-Food Industry Analytical Chemists Committee showed that the differences in the results using the three instruments were frequently less than 10% RSD.

P-88 Food Testing made Simple and Reliable – Fully Automated and Integrated Modular QuEChERS Sample Preparation

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The number of pesticides and metabolites being monitored in food commodities is well over 1'000 targeted analytes. Laboratories are being asked to broaden their range and increase the quality of pesticide analysis, while management is demanding cost reductions. QuEChERS, standing for Quick, Easy, Cheap, Effective, Rugged, and Safe is readily accepted by both the Association of Analytical Communities (AOAC) and the Committee of European Normalization (CEN) for the analysis of pesticide residues in food and agricultural products1. QuEChERS is a simple and straightforward sample preparation technique for multi-residues screening and has now been automated by Chemspeed. Multi-residue methods increase sample throughput, decrease sample turnaround time, and optimise analyst efficiency. The QuEChERS method is a two-step process: extraction followed by clean up. The key advantages are: high recoveries, accurate results, high sample throughput, low solvent and glassware usage, lower reagent cost, robustness and ruggedness.

P-89 High Throughput Screening of Food Contact Materials

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The use of the ASAP probe can substantially reduce the time of analysis, producing qualitative results and identification of potential migrants with increased confidence when used in conjunction with high resolution MS detection techniques, such as time-of-flight (ToF) MS. The use of ToF-MS also allows full scan screening of the samples so potential migrants other than those specifically analyzed for may also be detected. Most food and drink is packaged in some way. It is also highly likely that it comes into contact with other materials during harvesting, production, transport, storage, and cooking. A food contact material (FCM) is any material or article intended to be placed in contact with foodstuffs. Food packaging materials are the most notable example, but also included are cutlery, dishes and plates, containers, parts of food processing equipment, etc. Over the last couple of years there have been numerous notifications relating to the migration of PAAs from nylon kitchen utensils via the Rapid Alert System for Food and Feed4 (RASFF). As concerns to human health grow regarding these FCMs, quicker and easier methods need to be developed to screen for compounds in the current legislation. This poster will detail the analysis of nylon kitchen utensils for PAAs and will show how the latest advances in mass spectrometer probe design helps to achieve this goal.

P-90 QuEChERS Approach for Determination of Seven Pesticides Residues In Quinoa (Chenopodium quinoa WILLD) USING GC-µECD

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The quinoa is a pseudo cereal origin from the Andean and is an important protein source, their culture is spreading in the worldwide, and disease problems are not alien to this culture, chemicals applications are needed for this control. The QuEChERS sample

preparation was used for determination of 7 pesticides residues in quinoa. The method involved 1% acetic acid- ethyl acetate extraction solvent with MgSO4 and CH3COONa followed by dSPE cleanup with PSA sorbent. The analyses were carried out with GC–µECD (Agilent 7890 A) equipment an Agilent HP-5 30m x 0.32mm x 0.25 µm column was used. The method showed linearity ≥ 0,98 and the LOQs for the pesticides studied are between 0.01 to 0.1 mg kg-1.The method was validated using a blank sample of quinoa spiked at 0.08; 0.2 and 0.4 mg kg-1; the percentage of recoveries were 50 to 96 with CV% 1 to 11; 32 to 73 CV% 4 to 8 and 20 to 102 with CV% 4 to 13 respectively for each spiked level. The QuEChERS approach method showed low recoveries, for the pesticides: chlorotalonil, heptachlor, captan, endosulfan alpha, endosulfan beta, endosulfansulfate and dieldrin; matrix effect and inadequate for analysis and quantification by GC–µECD. Keywords: Food; Validation, Multiresidue.

P-91 QuEChERS Approach for Determination of Seven Pesticides Residues in Honey Using GC-µECD

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The honey produced by the bees from the nectar´s plants and sugary secretions of the bees; can contain agrochemical products used in agricultural management. To evaluate these residues the QuEChERS method for sample preparation was used for determination of 7 pesticides residues in honey. The method involved 1% acetic acid- ethyl acetate extraction solvent with MgSO4 and CH3COONa followed by dSPE cleanup with PSA sorbent. The analyses were carried out with GC–µECD (Agilent 7890 A) equipment; an Agilent HP-5 30m x 0.32mm x 0.25 µm column was used. The method showed linearity ≥ 0,99 and the LOQs for the pesticides studied are between 0.007 to 0.05 mg kg-1. The method was validated using a blank sample of honey spiked at 0.07; 0.2 and 0.4 mg kg-1; the percentage of recoveries were 85 to 128 with CV% 6 to 19; 85 to 125 CV% 2 to 12 and 75 to 119 with CV% 7 to 26 respectively for each spiked level. The QuEChERS approach method showed adequate for chlorotalonil, heptachlor, captan, endosulfan alpha, endosulfan beta, endosulfansulfate and dieldrin for analysis and quantification by GC–µECD. Keywords: Food; Validation, Multiresidue.

P-92 QuEChERS Approach for Determination of Pesticides Residues in Strawberries Using GC-µECD

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QuEChERS method for sample preparation was used for determination of 7 pesticides residues in strawberries. The method involved 1% acetic acid- ethyl acetate extraction solvent with MgSO4 and CH3COONa followed by dSPE cleanup with PSA sorbent. The analyses were carried out with GC–µECD (Agilent 7890 A) equipment an Agilent HP-5 30m x 0.32mm x 0.25 µm column was used. The method showed linearity ≥ 0,99 and the LOQs for the pesticides studied are between 0.007 to 0.05 mg kg-1.The method was validated using a blank sample of strawberry spiked at 0.07; 0.2 and 0.3 mg kg-1; the percentage of recoveries were 62.4 to 132,7 with CV% 2 to 12; 46,1 to 122.8 CV% 3 to 13 and 50,6 to 102 with CV% 3 to 11 respectively for each spiked level. The QuEChERS approach method showed adequate for the pesticides chlorotalonil, heptachlor, captan, endosulfan alpha, endosulfan beta, endosulfansulfate and dieldrin and efficient for analysis and quantification by GC–µECD. Keywords: Food; Validation, Multiresidue.

P-93 Rapid Analysis of Pesticide Residues Using DART-MS

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Ambient desorption ionization mass spectrometry (MS) is a rapidly growing area representing an attractive alternative to conventional analytic approaches. Recently introduced ionization techniques, such as direct analysis in real time (DART), desorption electrospray ionization (DESI), or atmospheric pressure solids analysis probe (ASAP), allow direct examination of various types of samples in the open atmosphere and at ground potential. Little or no sample treatment prior to analysis is required. Additionally, time-consuming separation of sample components, which is usually employed by chromatographic methods, can be omitted with ambient MS.
In this study, the potential of the DART-MS technique to screen and, possibly, to quantify the residues of the most common strobilurin fungicides in wheat, and of dithiocarbamate fungicides in fruits was explored.

P-94 Developing a Multi-Residue Pesticide Method for Testing Fatty Foods

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The increased demand for multi-class pesticides in a wide range of diverse food products has incentivized the development of methods to accommodate these needs. Traditionally, our laboratory has focused on multi-class pesticide screens for produce and grains. Recently, we have developed a streamlined version of S19 for multi-pesticide testing from fatty food products (e.g. dairy-based products, chocolate, nuts, etc.). The method has been designed to test for hundreds of pesticides from a single sample

preparation and single analysis. This method allows us to meets the needs of our customers and is cost and time efficient for our laboratory's operation.

P-95 Method Improvements for the Analysis of Glufosinate in Plant Matrices

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Glufosinate is a non-selective herbicide that is marketed under the Ignite and Liberty labels. Several Liberty-tolerant crops are in or entering the market (canola, corn, cotton, soybean), and the non-GMO label for this herbicide is being expanded as well. Due to these reasons, the acreage of glufosinate use could steadily increase, and more labs outside of Bayer will be called upon to test for this compound. The current enforcement method is time and labor intensive and uses older instrumentation for analysis (GC-FPD). Bayer has developed a newer method of analysis that has increased sample throughput, uses more common laboratory equipment, and is adapted to LC/MS/MS technology.

P-96 A LC/MS/MS Method for the Detection of Penflufen Residues in an Aquatic Dissipation Study using Rice Seeds Treated with Penflufen

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An aquatic field dissipation study was conducted using rice seed treated with penflufen, an experimental fungicide proposed for control of diseases in potato, wheat, barley, soybeans, canola, peanuts, cotton, dry beans, corn and sunflowers being developed by Bayer CropScience. The plot was planted with penflufen treated rice seeds and at the appropriate intervals over one year soil cores were collected for analysis. The procedure used to collect and analyze a representative soil sample to monitor the decline of penflufen and its metabolites over the duration of the study is described. Penflufen and its metabolites were extracted from the soil by microwave extraction/shaking, the sample amended with isotopically labeled internal standards, an aliquot centrifuged and analyzed by LC/MS/MS.

P-97 Fast Screening of Pesticides and Toxins Using Multiple Analytical Techniques at the Food and Drug Administration

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The Food and Drug Administration's pesticide residue program is advancing to meet the challenges to increase the speed and scope of its pesticide and toxin coverage. Using a targeted approach, pesticides are analyzed and quantitated against standards for over 240 compounds by LC-MS/MS in 15 minutes, and more than 200 pesticides by GC-MS/MS in 20 minutes. Each sample is additionally screened to detect 900 pesticides using full scan GC-MS coupled with spectral deconvolution against standardized MS spectra and retention indices. Looking to the future, FDA plans to expand pesticide and toxin coverage by an additional 500-600 compounds using LC coupled with high resolution mass spectroscopy by orbitrap. Also, under evaluation is a procedure to streamline import sample processing by prescreening pesticides and toxins on the surface of raw agricultural commodities using ambient pressure desorption with high resolution mass spectrometry at the port of entry.

P-98 Comparison of 4 ethyl acetate based methods and original QuEChERS for the analysis of pesticide residues in blueberries.

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Demand for blueberries has been increasing in recent years and fresh-market prices have been relatively high. Uruguay reported 100 ha of newly established plantings in 2004, increasing lately mainly for local consumption and high exportation. Fungicides are the most employed pesticides in blueberry crops and are used during flowering and fruit setting stages. Blueberry is an acidic matrix with high content of polyphenolic compounds like anthocyanins, proanthocyanidins, resveratrol, flavonols, and tannins. These compounds have similar solubility to the pesticides under study. In this work, four dispersive methodologies based on ethyl acetate extraction using borax Na2B4O7 10H2O:MgSO4 and NaCl:MgSO4 for the salting out step, with and without GCB for the clean-up were evaluated and compared to the original QuEChERS method. Recoveries, linearity, matrix effects and reproducibility were evaluated for eighteen fungicides with all the studied methods by GC–MS. Positive findings of fungicide residues in real samples are shown

P-99 Automated LC/MS Analyses of Pesticides and Mycotoxins Using QuEChERS and DPX Tips

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Cleanup of the extracts of fruit and vegetables was performed using QuEChERS tips. The QuEChERS tips were designed to extract just 0.25 mL of acetonitrile extracts, and the extractions were performed using a GERSTEL multi-purpose sampler (MPS) equipped with the DPX option. Following automated "cleanup", the extracts were injected and analyzed using an AB SCIEX 3200 Q-Trap. A mixture of representative polar pesticides, including organophosphate, organonitrogen and carbamates, were extracted and analyzed. Various types of sample matrices were analyzed, and recoveries were generally greater than 80% with less than 10% RSDs. In addition to pesticides, aflatoxins in grain products were also analyzed using Disposable Pipette Extraction (DPX) Tips. In this case, DPX-WAX (weak anion exchange) tips were found to provide high recoveries of aflatoxins (app. 90%) with good reproducibility (app. 10% RSDs).

P-100 Screening for Pesticides by LC/MSMS and GC/MSMS in Muscle Tissue

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A screening method is used for the extraction and detection of the various classes of pesticides in muscle tissues. Pesticides are extracted from muscle tissue with high speed dispersion in ethyl acetate followed by solvent exchange to acetonitrile and clean-up using ultra-low temperature freezing, centrifugation, and solid phase extraction. Detection of pesticide residues is performed by GC/MS/MS and LC/MS/MS. This pesticide screening method detects the highest risk pesticides identified by the EPA and more compounds will be added.

P-101 Large Volume Splitless Injection Using An Unmodified Splitless Injection Port For The Analysis of Halogenated Pesticides And Brominated Flame Retardants

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Although many halogenated pesticides and flame retardants are currently banned and have been phased out of production, they continue to be monitored due to their persistence in the environment and bioaccumulation in humans and animals. The United States Environmental Protection Agency has developed Method 527 for the determination of selected pesticides and brominated flame retardants in drinking water. This method recommends a splitless injection but allows for the use of alternative injection techniques such as large volume injections which typically require a special injection port. However, a relatively new technique describes the use of a typical splitless injection port for large volume splitless injections (LVSI) (Patent No: US 6,995,709 B2). This technique utilizes a fast autosampler injection onto a liner containing glass wool, a pre-column (e.g. 5m x 0.53mm) press-fitted to the analytical column, and a starting oven temperature below the boiling point of the solvent.

P-102 Method for the Analysis of 4-methylimidazole by LC/MS/MS

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4-methylimidazole (4MI or 4-MEI) is a by-product formed during the production of caramel colorings. These colorings are added to many foods including beverages (cola), coffee, beer, whisky, baked goods, soy sauces and meats. To date, studies have not provided a clear indication as to the toxicity of 4MI but the National Cancer Institute has nominated 4MI as a candidate for toxicity and carcinogenicity studies. In addition, the EFSA has recently changed the allowable 4MI levels in foods. The chromatographic analysis of 4MI has traditionally been accomplished by GC/MS with derivatization, RP-HPLC with ion pairing, and now HILIC. None of which are simple or easily reproducible. In this presentation we utilize our Ultra PFPP column to analyze 4MI using typical LC/MS mobile phases, water and methanol with formic acid, and isocratic conditions in ESI+. We also explore the retention mechanism of the PFPP phase that allows the retention of small polar compounds such as 4MI.

P-103 Comprehensive Screening for PAHs in Sugarcane Products after QuEChERS with Sweet Separations using an Rxi-17Sil MS column, GC-MS, and GCxGC-TOFMS

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Polycyclic aromatic hydrocarbons (PAHs) were determined in various sugarcane products using QuEChERS sample preparation, GC-MS with selected ion monitoring (SIM), and comprehensive two-dimensional GC with time-of-flight MS (GCxGC-TOFMS). In sugarcane production, it is common to set fields on fire to make harvesting easier by burning the leaves while leaving the sugarcane stalks intact. Unfortunately, sugarcane burning can produce toxic PAHs. Although a variety of sugarcane products (e.g. juice, liquors) have been tested for the presence of PAHs, often the number of PAHs investigated has been limited. We tested sugarcane products including cachaça, other sugarcane liquors and sugarcane, for the presence of PAHs. QuEChERS-based sample preparation and separations on an Rxi-17Sil MS column were investigated with attention to extraction recoveries and chromatographic resolution of isobaric PAH compounds from EPA and EU lists, as well as others.

P-104 Pesticide Analysis for Organic Cut Roses using QuEChERS, GC-TOFMS, and GCxGC-TOFMS

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Currently, the United States Department of Agriculture (USDA) regulates only infestation and disease on imported cut flowers, representing approximately 70% of the rose market. This regulatory situation unintentionally encourages heavy use of pesticides, which are not regulated by USDA for cut flowers. Heavy pesticide use can have serious consequences for the health of workers in the flower industry, and their local environment. To address those issues, an organic flower industry exists that advertises agricultural sustainability practices that include limited pesticide use. In this contribution, the Quick – Easy – Cheap – Effective – Rugged – Safe (QuEChERS) sample preparation method was used with GC-TOFMS and GCxGC-TOFMS to analyze pesticides on cut roses. Pesticide classes selected for investigation include organochlorine, organophosphorus, carbamate, pyrethroid, and triazole compounds that are routinely used by the flower industry. Development and optimization of sample preparation, including extraction and cleanup, will be highlighted. Baseline residue levels for organic certified roses and non-certified roses will be estimated in hopes of determining whether "a rose is a rose is a rose".

P-105 The QuEChERS Tablet

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The QuEChERS sample preparation approach is by now well used and several commercial vendors provide pre-packaged extraction salts and dispersive solid phase extraction sorbents. Unfortunately, in some cases the extraction salts (magnesium sulfate, etc.) come in the 50 mL centrifuge tube used for extraction, which is inconvenient because the method specifies addition of homogenized sample and acetonitrile extraction solvent to that tube prior to salt addition. This necessitates a transfer of salts, from one tube to another, increasing the risk of leakage due to salts in cap threads, and possibly increasing plastic waste. We have been exploring the use of tablets for QuEChERS extraction salts to make the delivery of these salts even easier. A tablet allows an easy delivery of extraction salts to sample/acetonitrile in a centrifuge tube, and decreases the risk of spillage and leakage. This presentation will focus on initial results achieved for QuEChERS extractions with tablets, discussing features, benefits, and manufacturing difficulties.

P-106 A Comprehensive Approach to Pesticide Residue Monitoring, Including Non-target Analysis, for Fruits, Vegetables, and Nuts, Using QuEChERS, LC-MS/MS, and GCxGC-TOFMS

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Food pesticide residue monitoring has traditionally been performed using gas chromatography (GC), but there is increasing use of liquid chromatography (LC) with tandem mass spectrometry (MS/MS). With MS, complete chromatographic resolution is not essential, as selected ions are used for pesticide identification and quantification. However, data quality can be improved through better retention and separation. In GC, better separation can come from comprehensive two-dimensional GC (GCxGC), an approach involving two separations on orthogonal columns in one analytical run. The QuEChERS approach was used for red bell pepper, cucumber, lemon, raisins, spinach, hazelnuts, and red grapes with subsequent pesticide determinations by LC-MS/MS and GCxGC-TOFMS. Samples spiked showed good recoveries. While GCxGC-TOFMS was limited in the number of pesticides determined, versus LC-MS/MS, selectivity and sensitivity was enhanced by GCxGC. The full mass-range capability of TOFMS allowed non-target compound identification. LC-MS/MS was extremely sensitive and selective for the targeted compounds.

P-107 Evaluation of Elution Solvents for Three Solid-Phase Extraction to Predict Appropriate Clean-up Method by Matrix and log P Characteristics of Pesticides

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Three kinds of solid phase extractions (SPE) with florisil, NH_2 and C18 were evaluated using different solvents mixture to find the correlation with pesticide log P and solvent strength. Twenty five pesticides of various log P value were selected and analyzed by HPLC/UVD from fortified matrices such as rice, bean, pear and lettuce. Recoveries of pesticides in rice, pear, and lettuce were ranged 76.2~106.2% in three SPEs. But recoveries in bean was affected by oil content and were about 10% lower than others in florisil and NH_2 SPE. In normal phase with florisil SPE, almost of pesticides were eluted by the order of log P value. In NH_2 SPE, although some pesticides were retained longer and demanded stronger solvents than predicted, almost pesticides were characterized to specified elution solvents by log P range. C18 SPE of reverse phase was showed with much similar eluting order with expectation. The correlation

between retention time and log P was highly significant. When this relationship was expanded to 70 pesticides including 25 mentioned above using UPLC, the correlation was showed significantly as R^2=0.9362. From these results, proper solvents by SPE species could be selected considering log P value when new pesticide was analyzed.

P-108 Analyisis of Amitraz and 2,4-Dimethylaniline Residues in Colombian Honey by UFLC- MS

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A high percentage of apiaries in Colombia are concern about the Varroasis infection disease, so that some beekeepers around the country apply to their hives acaricides such as amitraz to control Varroa jacobsoni destructor. Amitraz may remain as residue in honey and also its metabolite 2,4-dimethyl aniline (DMA). The aim of this work was to study two varieties of the QuEChERS method approach for the determination of amitraz and DMA residues in honey by UFLC-MS. Three different buffers (acetate, citrate and carbonate) were compared based on the QuEChERS frame. All the methods lead to clean extracts. Among them, the AOAC method (acetate) showed the lowest percentages of recovery unlike the European method (citrate) that gave the best performance, showing values of (75 % and 90%) of amitraz and DMA, respectively. Both methods were performed at two concentration levels, 10 and 50 µg Kg−1 (n=4). The limits of quantification for the amitraz and DMA were 5 and 10 µg Kg−1, respectively.

P-109 Analysis of Pesticides in Celery Extract by UHPLC-TOF-MS

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Due to the health risk posed by pesticides consumption in food, the Food Quality protection Act (FQPA) in the US and the EU directive 91/414/EEC require low levels of pesticides in food. One of the limitations of studying pesticides by the traditional LC/MS/MS methods is the choices one has to make as to which pesticides to monitor during multiple reaction monitoring analysis. However in time-of-flight (TOF) mass spectrometry, one can record the entire spectral range during analysis and hence study both target and not-target analytes using the accurate mass capability of the TOF in a given sample. We present a study of pesticide analysis in a celery extract that was obtained by the QuEchERS method of food extraction. The celery extract was spiked with varying concentrations of a mix of 14 pesticides and analyzed by UHPLC-MS using the PerkinElmer FlexarTM FX-10 UHPLC pump with PerkinElmer AxION(TM) TOF as the MS detector. We could easily detect majority of the pesticides in the range of 0.98-3.9 µg/kg and in some cases detect as low as 0.1 µg/kg well within the EU LOD requirement range of 10 µg/kg. The presence of the target analytes was confirmed by retention time and mass accuracy measurements. The accurate mass of the [M+H]+ ion of each of the pesticides and a fragment ion if present was determined and observed to be < 2 ppm in most cases. In conclusion, the LC-MS-TOF can measure pesticides in food samples with LODs in compliance with EU requirements and shows excellent selectivity to unambiguously identify the target analytes in food.

P-110 One Step Extraction, Clean-up and concentration of Chlorinated Pesticides in Raw Coffee Beans

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Organochlorine pesticides are man-made chemicals once widely used in agriculture and for mosquito control. Beginning in the 1960's, public awareness of their toxicity began to increase and by the mid 1970's; many began to be banned from use in the United States. Though some are still registered for use in the United States, the vast majority of their usage exists in developing nations. Classified as persistent organic pollutants, organochlorine pesticides, they exhibit a wide range of toxic effects including reproductive and neurotoxicity, as well as liver and kidney failure. In animals, they have been shown to cause cancer in these organs. Due to their persistent nature, and toxicity, monitoring food sources for organochlorine pesticides is an increasing growing concern for both food vendors and consumers. Food sources typically being difficult matrices to extract and analyze, the following procedure outlines the extraction, extract clean-up and concentration of raw coffee beans for the detection of these compounds in one rapid and efficient process.

P-111 Modern Water Analysis:The Use of Modern Chromatography Coupled to In-Line Solid Phase Extraction (SPE) / ESI –Triple Quadrupole / Hybrid Linear Ion Trap Mass Spectrometry, for the Analysis of Surface, Drinking and Wastewater

Frederic Ciner and Ed Dabrea, **Jupiter Analytix**, 150 S. Old Dixie Highway, Jupiter, FL 33458; 561-575-0030, rciner@jupiterlabs.com

Our focused research and developed methodology involves the simultaneous Quantitation of Pesticides, Personal Care Products, Endocrine Disruptors, and other anthropogenic inputs into water systems at ultra trace levels using HPLC/MS/MS and In-Line automated SPE/HPLC/MS/MS. Currently our group is realizing low femtogram on column sensitivity without enrichment for many Drugs of Abuse, OP's, Pharmaceuticals, Pesticides and Endocrine Disruptors. This level of sensitivity is reached using modern stationary phase coupled to QQQ-Ion trap MS with Scheduled Multiple Reaction Monitoring (SRM). Dynamic Ranges cover 3 orders of magnitude and with LOQs that are in the single to 10 PPT range while demonstrating linearity (r^ > 0.995) and robustness.

NOTES

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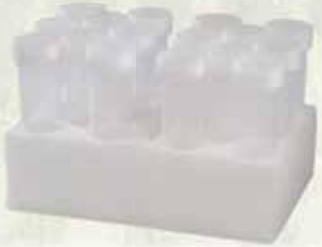
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