

VENDOR SEMINARS

Refreshments provided by the vendors

V-1 Sunday, 7:00 – 8:00 PM

Enhancing That Certain Part of the GC Anatomy with QuEChERS and Cartridge SPE Cleanup for Pesticides in Dietary Supplements

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

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

V-2 Monday, 7:00 – 8:15 AM

An Overview of Advanced MS Technologies for Residue Screening; UltraPerformance Residue Analysis using UPLC TQ MS; Capability of QToF for Target and Non-Targeted Screening in Food, Water and Wildlife Samples

Timothy Jenkins; **Waters Corp.** Chemical Analysis, Atlas Park, Simonsway, Manchester, England, M22 5PP, **UK**; +44 161 435 4100, timothy_jenkins@waters.com

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

V-3 Monday, 12:15 – 1:15 PM

EPA Method 1699: High Selective Multiresidue HRGC/HRMS Pesticide Analysis Applied to Food Samples

Heinz Mehlmann, Dirk Krumwiede, and Frank Theobald²; **Thermo Fisher Scientific**, Bremen, Hanna-Kunath Str. 11, Bremen, Bremen, 28199, **Germany**; 004942154930, heinz.mehlmann@thermofisher.com
2. Environmental Consulting Cologne, Cologne, **Germany**

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

V-4 Tuesday, 7:15 – 8:15 AM

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

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

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

V-5 Tuesday, 12:15 – 1:15 PM

Chemistry of Solid Phase Extraction

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

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

V-6 Wednesday, 7:15 – 8:15 AM

Keys to an Analytical Method: Focus on Mycotoxins

Wendy Rasmussen and Bill Holmes²; **Pickering Laboratories, Inc.**, Technical Sales Dept., 1280 Space Park Way, Mountain View, CA 94043; (650)694-6700, wendyr@pickeringlabs.com
2. Mississippi State University, Starkville, MS

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

V-7 Wednesday, 12:15 – 1:15 PM

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

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

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