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Washington Chromatography Discussion Group

the

Washington Chromatography Discussion Group

presents

the 3rd annual

Open House & Poster Session

September 18, 2013

6:00 pm - 8:00 pm

Bethesda North Marriott
Hotel & Conference Center
Salons B-C

http://wcdg.squarespace.com

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Dear Colleagues,

The Washington Chromatography Discussion Group (WCDG) thanks you for participating in the third annual WCDG Open House & Poster Session. On behalf of the group, I hope that you enjoy the science and networking opportunities that an event like this provides. The WCDG is composed of several hundred enthusiastic separation scientists in the Washington DC area with interests in separations techniques such as LC, GC, and CE. Our membership is composed of industry, government, and academic professionals with a goal of expanding our understanding in chromatography and its applications.

As your host for this event, I wish to welcome you to our Open House & Poster Session and to thank you for your participation.

Sincerely,

Jonathan Edelman
WCDG President

Advantages of Mass Spectrometric Detection for Polymers Separated by UltraPerformance Convergence Chromatography

Annie Landicho, Waters Corporation
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Coauthors: Oliver Burt, Baiba Cabovska, Michael O'Leary, Peter Hancock

Abstract:
Synthetic polymers are commonly analyzed by mass spectrometry to determine molecular weight distribution, chemical composition, produce supplementary information about individual oligomers and polymer chain length among other characteristics.

Separation of chemically similar components is critical. We investigated combining UltraPerformance Convergence Chromatography with MS using sub-2 um particle size stationary phases and a CO2-based mobile phase to take advantage of operating at reduced column temperature relative to GC for thermally labile components.

2013-2014 WCDG Board

Future WCDG Meeting Dates

The WCDG holds regular meetings, generally on the third Wednesday of each month from September through May at the US Pharmacopeia in Rockville, Maryland. (Some variability in day and location) At 6:00 p.m. a light dinner is served followed by the featured speaker at 7:00 p.m. We welcome you to join in our discussions. Visit our website (http://wcdg.squarespace.com) for updates and additional information.

Tentative Meeting Dates and Locations for 2013-2014

Wednesday, October 23, 2013
US Pharmacopeia, Rockville, MD

Wednesday, November 13, 2013
US Pharmacopeia, Rockville, MD

Wednesday, December 18, 2013
US Pharmacopeia, Rockville, MD
(Joint with the Chemical Society of Washington)

Wednesday, January 22, 2014
US Pharmacopeia, Rockville, MD

Wednesday, February 12, 2014
TBD

March 2014
Chicago, IL

Monday, April 14, 2014
Shimadzu Training Center, Columbia, MD
(Joint with WBMSDG)

Wednesday, May 21, 2014
US Pharmacopeia, Rockville, MD

Advances in Particle and Packing Technology for Higher Efficiency Ultraperformance LC Columns

Donna Zwirner, Waters Corporation
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Coauthors: Thomas Walter, Kevin Wyndham, Stephen Shiner, Daniel Walsh, Babajide Okandeji, Michael Savaria, Pamela Iraneta, Jonathan Turner, Cheryl Boissel, Scott McCall

Abstract:
The pursuit of higher efficiency liquid chromatography (LC) columns has occupied many chromatographers over the last forty years. The primary approach used to increase efficiency per unit length has been to decrease the size of the particles in the column. In addition to particle size, particle morphology (fully porous (FP), solid-core (SC) or nonporous) has been shown to have a significant effect on efficiency. While notable increases in efficiency have been demonstrated for 4.6 mm internal diameter (i.d.) columns packed with 2.6/2.7 µm SC particles relative to columns packed with similar size FP particles, comparable increases have not been previously demonstrated for 2.1 mm i.d. columns packed with < 2 µm SC particles. Columns of 2.1 mm i.d. containing < 2 µm particles are preferred for UltraPerformance LC (UPLC®) because they offer optimal speed, sensitivity and resolution together with low solvent consumption.

We have developed 2.1 mm i.d. UPLC columns packed with 1.6 µm solid-core particles that show dramatically increased efficiencies. These higher efficiencies are achieved without a significant increase in back pressure compared to columns packed with 1.7 µm FP particles. In combination with novel bonding chemistries, this improved particle and packing technology produces columns that show outstanding performance for separations of a range of analytes, from small molecules to peptides.

Ultrasensitive Quantification Assay for Oxytocin in Human Plasma using a LC/MS Microfluidic Platform

Ann Gray, Waters Corporation
Milford, MA
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Abstract:
Quantitative LC/SRM-MS methods are used routinely for providing bioanalytical support for small-molecule drugs in various stages of drug discovery and development. There is growing interest in developing bioanalytical methods for the quantification of therapeutic peptides and proteins in serum/plasma to support preclinical and clinical pharmacokinetic/pharmacodynamic (PK/PD) studies. Ligand binding assays (e.g., ELISAs) have been historically used for measuring protein concentrations in biological fluids, but these assays suffer from major drawbacks such as lengthy assay development time, poor reproducibility, and inaccurate results due to cross-reactivity.

Here we report an ultrasensitive (1 pg/mL LLOQ) capillary flow LC/SRM-MS assay for oxytocin in human plasma, able to provide the same desirable features that are typically incorporated in small-molecule quantification: high specificity, robustness and high-throughput.

WCDG Membership

Are you interested in becoming a member of the WCDG? Membership dues are only $10 per year (Sept-May) and can be paid to the Treasurer, Al Del Grosso, at any meeting.

Dues can also be paid online at any time at http://wcdg.squarespace.com (find the "Pay Now" button on the right menu bar)

WCDG Mailing List

To be added to the WCDG mailing list, visit our website at http://wcdg.squarespace.com (find the "Subscribe" button on the right menu bar) or email washchrom@gmail.com.

WCDG Corporate Sponsorship Program

Are you interested in becoming a corporate sponsor of the WCDG? We offer many levels of corporate sponsorship to fit the needs of any chromatography company, big or small. Visit our sponsorship site at http://wcdgsponsorship.ezregister.com for more information about the sponsorship levels and to join our growing corporate sponsorship program.

Questions about sponsorship? Contact board members Jonathan Edelman (jonathan.edelman@gmail.com) or Mallory Morris (mjharlow09@gmail.com) for more information.

WCDG Programming

Do you have program or speaker ideas? To make suggestions or offer to give a presentation to the WCDG, contact the Program Chair, Claire Chisolm, at cnc@usp.org.

Detection of Low Levels of Estradiol with the Use of Micro Flow LC Coupled to a Sensitive MS System

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Coauthors: Alexandre Wang; Jesse Seegmiller; Witold Woroniecki; Hua-fen Liu

Abstract:
Estradiol is an important biomarker and an analytical method capable of detecting low pg/mL concentration is essential in the study of this steroid. In this approach, we take advantage of the reduced flow rate from a micro LC system combined with a highly sensitive mass spectrometer system in order to reach the desired limit of detection. The use of a micro flow rate allows not only for more efficient ionization and sampling but also for significant cost savings with the reduction in mobile phase consumption.

Quantitative Capabilities of Two-Dimensional Gas Chromatography (GCxGC) and Liquid Chromatography (LCxLC) for the Analysis of Standard Reference Materials

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Co-authors: Jacolin A. Murray, Mallory J. Morris, Melissa M. Phillips, Catherine A. Rimmer, Lane C. Sander

Abstract:
Two-dimensional chromatography is an excellent technique for the separation of analytes from other co-eluting analytes and/or sample matrix interferences. Early work in both two-dimensional gas chromatography (GCxGC) and liquid chromatography (LCxLC) mainly focused on qualitative analysis. Quantitative methods were slower to emerge due to the lack of adequate data processing methods. Two-dimensional chromatographic data requires different approaches to handle and process (e.g. chromatogram display peak identification and integration) than single dimension chromatographic data. There are commercially available software packages for many of these functions and several were used to process the data. In addition a program for the visualization and data processing of two-dimensional chromatograms was developed in-house using R statistical software an open-source program. This program allows for visualization peak finding and peak integration parameters to be adjusted quickly in order to identify their impact on quantitation and to automate the data processing procedure. The different approaches for the processing of two-dimensional chromatographic data will be presented. Also multiple analytical methods were developed using both GCxGC and LCxLC with mass spectrometric and spectroscopic detectors for the analysis of compounds in various Standard Reference Materials (SRMs) available from the National Institute of Standards and Technology (NIST). The quantitative capabilities of the two-dimensional analytical methods for SRM measurements will be discussed.

Analysis of Flavor Chemicals by Simultaneous GC-MS and GC-FID

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Abstract:

The Department of the Treasury's Alcohol and Tobacco Tax and Trade Bureau (TTB) is responsible for regulating the use of ethanol in products manufactured in the United States, and collecting revenue generated from such use. One area that falls within TTB's jurisdiction is the regulation of nonbeverage products. Nonbeverage products are medicines, medicinal preparations, food products, flavors, flavoring extracts, and perfumes which are manufactured using tax-paid distilled spirits, and which are unfit for beverage purposes. One such way to ensure that products are unfit for beverage purposes is for the manufacturer to add specific flavor agents to the ethanol base at levels determined by TTB.

A quantitative method was previously developed to determine the concentration of various chemicals commonly used in flavors using gas chromatography (GC) coupled to both a mass spectrometer (MS) and a flame ionization detector (FID). The focus of the present study was to determine if additional compounds of interest could be successfully quantitated utilitzing the same acquistion parameters. Target analytes studied included (-)-Borneol, 1-Hexanol, 1-Heptanol, 1-Octanol, 2-Octanol, 1-Octen-3-ol, 1-Nonanol, cis-6-Nonen-1-ol, 1-Decanol, 1-Dodecanol, Geraniol, and (E)-trans-Isoeugenol. The internal standard (ISTD) used for quantitation was 2-nonanol. Accuracy was determined, with recoveries above 95% for most of the compounds at the three levels tested.

Separation Improvements with orthogonal chemistries and 2D LC using superficially porous columns

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Abstract:

The analysis of impurity is an important part of the development process. The fact that impurities are similar in structure can pose a separation challenge. Of particular interest are early eluding polar compounds, as they are often biologically active. A solution to this challenge can be to use 2D LC with heart cutting experiment where the sample is separated on a first column then elated into a loop, then the sample is transferred to a second dimension column by way of a valve. In this way co-eluding peaks can be resolved. In this experiment a mixture of sulfa drugs were used to separate structurally similar compounds by 2D LC.

Heart-Cutting Two Dimensional LC with QTOF Detection for the Determination of Peak Purity of Forced Degraded Insulin

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Coauthors: Dawn Stickle and Bob Giuffre

Abstract:
One critical aspect of drug safety involves determining suitable storage conditions for biological products by conducting stability studies under accelerated storage conditions. Prior to beginning a stability study one must develop stability indicating analytical methods or methods capable of detecting potential changes that occur during the study. In order to determine if the analytical method is stability indicating, the drug substance and drug product are subjected to forced degradation e.g., acid, base, oxidation and light. The main drug peak is then evaluated for peak purity. Typically the analytical method that indicates the impurity profile of a drug and that can be proven to be stability indicating is a HPLC method using UV detection. Often times the peak purity is determined by a comparison of UV spectra or by examining the peak using mass spectrometry. Although these techniques are effective at determining peak purity, they are not 100% reliable as low level impurities still may be not be detected under the main peak for various reasons, for example many impurities can have similar UV spectra to the main peak or ion suppression in mass spectrometry. Another approach to determine peak purity would be to use a heart-cutting two dimensional LC technique, in which the main peak would be sent to a second HPLC column and subjected to further separation. If impurities are present under the main peak, the second dimension LC column can likely separate these impurities from the main species. Historically two dimensional LC techniques are not in routine use as they can be difficult to construct and troubleshoot. In this work, we will present a robust and easy to use heart-cutting two dimensional LC attached to a QTOF mass spectrometer detector for the peak purity determination of base degraded insulin.

Determination of Ceftiofur Metabolite Desfuroylceftiofur Cysteine Disulfide in Bovine Small Intestine by LC-MS/MS

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Coauthors: Shixia Feng, Oscar A. Chiesa

Abstract:
The objective of the present study is to develop and validate a fast, specific LC-MS/MS method to determine the concentration of desfuroylceftiofur cysteine disulfide (DCCD), an active metabolite of ceftiofur in duodenum, jejunum, and ileum of the bovine small intestine. Ceftiofur is a cephalosporin beta-lactam antibiotic that is approved in the United States for treating respiratory diseases in cattle and mastitis in lactating dairy cattle. An accurate assessment of the concentration of an antimicrobial agent in the small intestinal tissue may be useful in predicting the potential emergence of antimicrobial-resistant bacterial pathogens. Eight steers were dosed with ceftiofur sodium at 2.2 mg/kg, IM at 24 hour intervals for 5 days. Different sections of the small intestine were collected from one steer each harvested at 12, 24, 30, 36, 48, 60, 64, and 76 hrs after the last dose. Stable isotope labeled d3-DCCD was used as an internal standard (IS). The analyte and IS were extracted from sample tissues by precipitating proteins using 1% formic acid in acetonitrile. The sample extract was cleaned up by solid phase extraction. The sample extracts were centrifuged and the supernatants were chromatographically separated with Kinetex C18 column and analyzed by triple quadrupole tandem mass spectrometry. The calibration curves were linear over a wide range of concentrations. Independent sources of control small intestinal tissues were used for the determination of accuracy and extraction efficiency. The average inter-day accuracy met the acceptance criteria according to the FDA guidance (GFI #145). The detectable concentrations are reported at harvest time points at 24 and 30 hrs after the last dose only from ileum tissues. The concentrations of DCCD in the gut tissue were relatively higher than the plasma concentration. This finding necessitates considering a potential risk evaluation procedure for the possible emergence of antimicrobial resistant bacterial pathogens from food animals.

Certification of Creatinine in Standard Reference Material 3667 Creatinine in Frozen Human Urine by Liquid Chromatography-Mass Spectrometry

Johanna E. Camara, NIST
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Coauthors: Karen W. Phinney

Abstract:
Background: Creatinine levels in serum and urine are important indicators of kidney function. In addition, other analytes are often normalized to creatinine levels to adjust for urine sample volume variation and very low levels of creatinine in urine may be indicative of sample adulteration. The objective of this study was to assign a certified creatinine value to a new Standard Reference Material® (SRM) provided by the National Institute of Standards and Technology (NIST) in order to support accurate creatinine measurements for the clinical community. SRM 3667 Creatinine in Frozen Human Urine was prepared from a pool of normal human urine of both males and females and contains an endogenous, unmodified level of creatinine. Methodology: To determine the linearity of the method, stock solutions of SRM 914a Creatinine and creatinine-d3 were mixed in appropriate amounts to produce calibrants with mass:mass ratios spanning 0.06 to 1.4 in 0.01 mol/L HCl solution. Evaluation of method accuracy was performed by spiking known amounts of SRM 914a creatinine into urine at three different levels. For certification analyses, SRM 3667 samples were prepared on two separate days (n=36 and n=24, respectively). Urine was combined with internal standard solution to achieve a 1:1 creatinine:creatinine-d3 mass ratio. The sample was then brought to a final 1:10 (volume fraction) dilution and a final 0.01 mol/L HCl concentration and allowed to equilibrate overnight. Prior to analysis, urine samples were diluted with additional 0.01 mol/L HCl to achieve a final 1:100 (volume fraction) concentration compared to original samples. SRM 967a Creatinine in Frozen Human Serum was analyzed as a control material. All calibrants and samples were separated by reverse-phase liquid chromatography (LC) using an isocratic gradient and detected by mass spectrometry (MS) in positive, electrospray ionization mode using single ion monitoring (SIM). The possible interferent creatine was also monitored by SIM. For additional validation, SRM 3667 was analyzed for creatinine by three external laboratories utilizing traditional chemical and enzymatic methods. Results: This method displayed linearity over the entire calibrant range (0.05 mg/dL to 1.0 mg/dL) with R2=0.9988. The % recovery was 104 % to 105 % for each spiked level of creatinine. The mean values for Day 1 and Day 2 were 614 µg/g and 612 µg/g, respectively, with within-day and overall % CV values ≤ 1 %. The final certified value ± expanded uncertainty for creatinine in SRM 3667 was reported as 613 µg/g ± 13 µg/g (61.8 mg/dL ± 1.3 mg/dL). Creatinine values provided by external laboratories ranged from 56.3 mg/dL to 67.8 mg/dL. Conclusion: This LC-MS method possesses appropriate linearity, accuracy, and precision to be utilized in the assignment of a NIST certified value for creatinine in SRM 3667. The creatinine value obtained by LC-MS analysis is comparable to values obtained by traditional chemical and enzymatic methods from external laboratories. SRM 3667 Creatinine in Frozen Human Urine will support accurate measurements of creatinine in urine by the clinical community.

Use of Schlieren photography to optimize source condition on ambient ionization: Direct source Analysis

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Coauthors: Joshua A Wilhide, William LaCourse

Abstract:
Schlieren photography is a technique used for studying the flow of fluids of varying density within an environment. Although this method is mainly used in the area of aerodynamics, it can prove useful when trying to visualize the ionization process of a Direct Sample Analysis (DSA) source. This source, commercially available from PerkinElmer, is still relatively new to the realm of mass spec and tuning for optimal source parameters for various different applications could prove time consuming. The goal for this project is to shed light on how different conditions within the source affect the overall efficiency of ionization for various applications. This project will also help correlate optimum source conditions to the chemical properties of various samples.

Images were taken using either a Canon PowerShot A620, Nikon D 3100 or Pentax K10 digital camera. The light source used was a very bright LED flashlight, partially covered to only allow a pinhole amount of light through. The mirror used was a concave spherical mirror, 150mm in diameter with a focal length of 1500mm. The camera was placed at twice the focal distance of the mirror with the DSA placed between the mirror and the camera. The flashlight was mounted on a ring stand and aimed at the mirror, bouncing the light back through the source region to the camera.

This seemingly unusual technique of Schlieren photography will be used to study how source conditions, including dry gas flow rate and temperature, nebulization pressure and temperature, source voltage, angle of DSA gun in relation to the inlet of the mass spectrometer and sample, as well as the size of orifice of the DSA gun. Work has been performed for proof of concept to show that Schlieren photography can be used to visualize the stream of gasses leaving the DSA gun and heading towards the sample. The first sets of images where completed with no sample introduction with factory standard conditions appropriate for adequate ionization efficiencies for most applications. This shows a dense stream of gas emanating from the source. Once the gas hits the inlet distortion of the gas stream occurs. The next sets of images were meant to study the effects of sample introduction on the gas stream and subsequent ionization. These images showed how the placement of the sample within the line of gas becomes a huge factor in how well the sample is introduced into the mass spectrometer. If the sample is not touching the stream, it will of course not ionize, but if the sample is placed halfway or more within the diameter of the stream, the distortion of the flow of gas will prevent most gas/ions from reaching the source and thus loss of signal is visualized. Additional images have shown how the drying gas, usually used to help with desolvation in ESI sources, becomes mostly a hindrance with DSA samples due to the distortion of the gas stream emanating from the source. Further work will be performed to improve image quality as well as expand on the source parameters examined.

Easy Pesticide Isolation & Concentration System with LC-MS/MS

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Coauthors: Stefan Kittlaus; Julia Schimanke; Günther Kempe; Marion Hoch; Karl Speer

Abstract:
EPICS is the first fully automated matrix separation technique for an improved pesticide analysis using LC-MS/MS. In comparison to the QuEChERS method EPICS does not require any time-consuming sample preparation steps. Pesticide residues are simply analyzed from an acetonitrile raw extract with high reproducibility.

Three novel Size Exclusion Chromatography columns designed for the Separation of a monoclonal antibody monomer from its impurities

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Abstract:
Monoclonal antibody is widely being used in the field of bio therapeutics. Separation of the pure antibody monomer needs to be very well resolved from its dimer and higher aggregates. Similarly for quality control and regulatory purpose the separation of antibody fragments is also very much essential. The species other than the monomer might induce toxic side effects to the body if not removed. We have developed three silica-based SEC columns designed especially for mAb analysis: 1. A 4.6 mm ID × 15 cm semi-micro column packed with 25 nm pore size, 4 μm particles, which is designed for high throughput analysis of mAbs in half the analysis time compared to the conventional TSKgel G3000SWXL, 5 μm, 7.8 mm ID x 30 cm SEC column. 2. A 7.8 mm ID × 30 cm analytical column packed with the same particles as mentioned above. The column dimension is compatible with conventional LC systems with relatively large extra-column dead volume and is suitable for high resolution analysis of mAb monomers and dimers. 3. A 7.8 mm ID × 30 cm analytical column packed with newly developed 30 nm pore size, 3 μm particles. Larger pore size with the estimated exclusion limit of ~4 x 106 Da provides improved separation and quantitation of mAb aggregates and oligomers. Here we report the features of these new SEC columns and their superior performance of mAb separation in comparison to conventional columns. We also report the efficient separation of aggregates induced by heat denaturation and acid denaturation using these columns, the separation of fragments generated by the digestion of the antibody by papain. This study shows that these 3 novel SEC columns are suitable for the analysis and purification of antibodies from their impurities.

Development of an Improved Standard Reference Material for Vitamin D Metabolites in Serum

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Abstract:
Studies have suggested that vitamin D has roles beyond musculoskeletal health and that deficiencies may lead to an increased risk of cancer, multiple sclerosis, and other serious diseases. Therefore, research continues into identifying optimal levels of vitamin D exposure. Interpretation and comparison of clinical data as well as population surveys has remained challenging because of poor comparability among the various assay methods used. In 2009, NIST introduced Standard Reference Material (SRM) 972 Vitamin D in Human Serum. This SRM was the first reference material available to evaluate the accuracy of analytical methods for 25(OH)D. The supply of this material was rapidly depleted, and recently NIST initiated development of the renewal material, SRM 972a Vitamin D Metabolites in Human Serum. Development of SRM 972a included improvements in both material preparation and value assignment when compared to the previous material.

Separation of fatty acid methyl esters by two dimensional gas chromatography

Pierluigi Delmonte, FDA
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Coauthors: Ali Reza Fardin-Kia, Douglas G. Hayward, Jeanne I. Rader

Abstract:
Oils extracted from products of marine origin such as fish and algae contain a large number of unsaturated fatty acids (FA) that can only be partially resolved by applying methodologies based on mono-dimensional gas chromatography (GC). Comprehensive two-dimensional gas chromatography (GC x GC) provides improved separation of FA (as methyl esters, FAME) by combining the selectivity of two GC separation columns. The analytes are separated on a plane instead of on a single dimension time axis. In this study, FAMEs are separated by an SLB-IL111 ionic liquid capillary column, reduced to their fully saturated forms by passing through a palladium reducer, and then re-separated by a shorter SLB-IL111. The application of the GC x online reduction x GC design provides the separation of FAMEs with different chain lengths, eliminating the most common cause of chromatographic overlapping when using highly polar separation columns. FAMEs are identified by GC x GC-TOF after their conversion to dimethyl oxazoline derivatives (DMOX). The palladium reducer and the second dimension separation column are replaced by a short BPX-35 column and FAMEs are indentified based on the distinctive mass spectra of their DMOX derivatives.

Evaluation of an Automated Method for the Fractionation of Extractable Petroleum Hydrocarbons (EPH) from Water and Soil Extracts

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Co-authors: Toni R. Hofhine[1], Noah Iskandarani[1], Patrick Sullivan[2]

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Abstract:
Government agencies and other regulatory bodies have developed several methods for determining the types of aliphatic and aromatic hydrocarbons that may be found in contaminated soil and water. One such method was developed by the Massachusetts Department of Environmental Protection and is often referred to as the MADEP EPH Method. This method utilizes a solvent extraction step followed by a silica gel fractionation into two extracts—an aliphatic extract (C9–C18 C19 –C36) and an aromatic extract (C11–C22). The two extracts are then concentrated and separately analyzed by gas chromatography with a flame ionization detector (GC/FID). The silica gel cleanup and fractionation step of the method requires a great deal of care and attention to detail to achieve satisfactory results. The purpose of this study was to evaluate an automated protocol for the fractionation of extractable petroleum hydrocarbons (EPH) into aliphatic and aromatic fractions using a Gilson GX-274 ASPEC™ System with Biotage ISOLUTE® Silica Gel Cartridges. Recoveries of aromatic hydrocarbons ranged from 86% to 97%. Recoveries of aliphatics ranged from 91% to 114%. No aromatics were observed in the aliphatic fraction. Automation of the fractionation process improved day-to-day precision and increased sample throughput compared to using manual fractionation.

Determination of furan in irradiated fruit and vegetables by headspace gas chromatography/mass spectrometry

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Abstract:
This presentation will describe the use of gas chromatography/ mass spectroscopy using static headspace sampling for the determination of furan in a variety of irradiated fresh fruit and vegetables and dried fruit. Furan is a suspected human carcinogen that has been found to form in some processed foods at low ng/g levels. Recent improvements in analytical methodology and scientific instrumentation have made it possible to accurately measure the amount of furan in a wide variety of foods. The survey results from the analysis of irradiated foods will be presented. In general no furan (less than the LOD) was detected in the non-irradiated fresh fruit and vegetables. However most of the dried fruit did have furan in them prior to the irradiation treatment. Furan was found at concentrations ranging from non-detectable (LOD 0.2-1.2 ng/g depending on the food matrix) to over 20 ng/g for foods irradiated up to 5 kGy. The level of furan for most of the irradiated fresh vegetables was less than the LOD. For the fresh fruit the concentration of furan ranged from non-detectable for pears to 14 ng/g for grapes. For dried fruit the concentrations ranged from 2 ng/g for apricots to over 20 ng/g for raisins cranberries cherries and blueberries. The concentrations of furan were higher for the dried fruit when expressed as ng of furan per g of fruit compared to the fresh fruit. This could be explained by the low water content of the dried fruit which would mean that more material was actually measured as well as the fact that the dried fruit had furan present prior to the irradiation treatment. The data demonstrates furan is generated in a variety of food products as a result of ionizing radiation but at the low part-per-billion concentrations.

Spiral Countercurrent Chromatography: Useful Separation for compounds

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Coauthors: Thomas M. Finn

Abstract:
A method of liquid-liquid partitioning chromatography is countercurrent chromatography carried out in centrifuged coils of tubing held in a spiral tubing support rotor. The separation rotor design and development will be described with examples of separations of small molecules peptides and proteins. The technology is licensed out of the NIH and progress in applications in many fields of chemistry is going on.

Application of liquid chromatography coupled mass spectrometry for quantification of target molecules

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Abstract:
Our study will introduce the combination of liquid chromatography (LC), multiple reaction monitoring mass spectrometry (MRM) and isotope-labeled internal standards for quantification of various target molecules including big molecules like proteins and peptides, and small molecules such as drugs. Our method enabled the investigations for quantification of membrane-associated proteins of low abundance and low solubility, characterization of specific isoform of biomarker proteins, multiplexing quantitative assay of multiple target proteins etc, suggesting the great potential of LC MRM in biomarker development and clinical diagnostics.