17TH Multidimensional Chromatography Workshop

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Abstract Book

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KL-1

THE GC×GC EFFECT: TRANSFORMING INDUSTRIES ONE MOLECULE AT A TIME

Haleigh Boswell

Chevron, Richmond, USA

Abstract

The complexity of petroleum matrices demands analytical techniques that transcend conventional methods. Comprehensive two-dimensional gas chromatography (GC×GC) has emerged as a transformative solution, overcoming key limitations of one-dimensional gas chromatography (1DGC) and standardized ASTM methods. GC×GC dramatically enhances peak capacity and resolution, enabling separation of thousands of compounds and improving detection of low-concentration analytes. Its structured chromatograms and superior spectral quality reduce false identifications, making it indispensable for analyzing complex mixtures.

However, the richness of GC×GC data introduces interpretive challenges—extracting actionable insights from massive datasets is not trivial. Automated workflows and in-house scripting further streamline data processing, empowering cross-functional teams to translate analytical depth into operational impact.

Having seen firsthand how GC×GC bridges the gap between molecular insight and industrial application, I will share my experience as an industry GC×GC expert on how this technique has enhanced our understanding of petroleum matrices at the molecular level. Drawing from professional experience, I will offer generalized insights that reflect broader analytical trends and personal observations. All examples and interpretations are intended to illustrate the wider value of GC×GC in petroleum analysis and do not reference company-specific data or proprietary findings.

KL-2

2D-LC AS A POWERFUL TECHNIQUE IN THE PHARMACEUTICAL SCIENTIST'S TOOLBOX FOR SMALL MOLECULES AND BIOLOGICS

Zach Breitbach, Michael Lesslie, Corianne Randstrom, Amanda Sulsberger, Lewis Meads, Chad Pickens, Jim Marren, Siqi Du, John Lawler, Andrew Koski

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Abstract

Multi-dimensional chromatography can be used in a variety of pharmaceutical applications in which a single, traditional chromatographic run does not provide sufficient information about one or more analytes. To enhance this separation, diverse run conditions or orthogonal columns and/or chromatographic modes can be used in the second dimension to isolate and reanalyze individual peaks in a single run. There are several applications where 2D-LC provides solutions to pharmaceutical related chromatographic challenges such as:

1. **Performing achiral and chiral analysis in a single run.** By performing chiral separation in the second dimension, all other impurities are essentially removed from the analysis, reducing the specificity requirements. Additional impurity information is obtained in the first dimension, reducing overall analysis time.
2. **Tracking unknown peak retention times when methods are modified.** To support the development of a bilayer fixed-dose combination tablet, unknown peaks were tracked from the established method for one of the drug substances onto a proposed method for the combination with the other drug substance.
3. **Identifying unknown peaks by mass spectrometry when non-mass spec compatible mobile phases or matrices exist.** This was applied for the peak identification of several phosphate prodrugs and their degradants, providing a facile approach as opposed to developing an additional method or isolating the impurities of interest.
4. **Enhancing confidence in specificity of methods by chromatographically analyzing peak purity.** A routine process is now being established at AbbVie and this new approach was applied to several drug substances.
5. **Enabling dual-assay methods for biologics.** An antibody-drug conjugate was analyzed for aggregates using SEC in the first dimension followed by reversed-phase characterization of the free-drug in the second dimension.
6. **Comprehensive 2D-LC for fingerprinting complex pharmaceuticals.** SEC x RPLC comprehensive 2D-LC was developed to fingerprint a natural mixture of > 400 small-molecules and enzymes present in a drug substance derived from natural sources.

To summarize, 2D-LC has been employed to solve non-routine analytical challenges in the pharmaceutical industry. It is anticipated that 2D-LC will continue to provide high-value information and solutions to analyze our increasingly complex drug portfolio.

KL-3

MULTIDIMENSIONAL CHROMATOGRAPHY FOR CHEMICAL ANALYSIS &NDASH; FROM SMALL MOLECULES TO SYNTHETIC POLYMERS

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Abstract

As society increasingly demands materials with safer and more sustainable profiles, the detailed characterization of complex chemical mixtures becomes crucial in new product development, material life-cycle analysis, and evaluating their environmental impact.

Multidimensional chromatography has proven to be a highly effective technique for chemical analysis. When coupled with universal and information-rich detectors such as high-resolution mass spectrometers, it provides more comprehensive chemical composition information, leading to new insights in material design and process development. This presentation will discuss several industrial applications that demonstrate how practical solutions utilizing GC×GC and LC×LC can address challenges in the analysis of recycled materials and complex polymer mixtures, spanning a wide range of molecular weights from small molecules to macromolecules.

KL-4

MULTIDIMENSIONAL GAS CHROMATOGRAPHY FOR PLASTIC WASTE PYROLYSIS

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Abstract

Accurate characterization of pyrolysis products is essential for developing reliable kinetic models and advancing both non-catalytic and catalytic plastic recycling. This presentation will first compare one-dimensional gas chromatography (GC) with two-dimensional gas chromatography (GC×GC) for analyzing complex pyrolysis oils. 1D-GC often cannot resolve overlapping mixtures of olefins, paraffins, and aromatics, limiting mechanistic interpretation. In contrast, GC×GC provides orthogonal separation and substantially enhanced resolution, identifying up to 5 times more compounds across various pyrolysis products. This comparison underscores why GC×GC is essential for comprehensive product deconvolution and accurate assessment of reaction pathways.

Building on this analytical capability, I will then demonstrate how coupling micropyrolysis with GC×GC–FID/TOF-MS enables verification of intrinsic-kinetic conditions for non-catalytic pyrolysis. Using design of experiments (DOE) and multivariate data analysis, we quantified the effects of particle size, sample size, temperature, and carrier gas flow rate on primary and secondary reactions.

Finally, we extend this framework to catalytic co-pyrolysis by integrating GC×GC with split-gas capture for simultaneous quantification of light gases and higher hydrocarbons. Using HZSM-5, we show that plastic mixtures, such as LDPE and PET, exhibit distinct optimal catalytic operating conditions and synergistic behaviors, emphasizing the importance of polymer-specific process design.

By leveraging the advanced separation capabilities of GC×GC, this talk highlights its critical role in elucidating both catalytic and non-catalytic processes and reaction mechanisms, thereby enabling resilient plastic-recycling solutions by deepening our understanding of pyrolysis chemistry, ensuring process adaptability, and reinforcing the foundations of a strong economy.

O-1

DISCOVERY-BASED ANALYSIS FOR TWO-DIMENSIONAL GAS CHROMATOGRAPHY TRENDS USING ALTERATION ANALYSIS AND TWO-DIMENSIONAL CORRELATION ANALYSIS

Christopher Freye, Matthew Herman

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Abstract

Two-dimensional gas chromatography (GC×GC) when coupled with mass spectrometry (MS) especially high-resolution mass spectrometry (HRMS) provides the ideal technique to discover minute chemical changes. However, GC×GC analyses result in extremely large datasets (GBs of information) wherein the statistically significant chemical changes are buried within the background chemical matrix. To combat these issues, many chemometric techniques have been developed or applied. Recently we introduced a new chemometric technique for GC-MS termed alteration analysis (ALA) and two-dimensional correlation (2DCOR) which can be used to discover statistically significant chemical changes across a series of chromatograms and understand the relationship between the statistically relevant changes. ALA generates three sets of information, the basic alteration map (BAM) which is the magnitude of the change, the synchronous alteration map (SAM) which describes the linear change, and the asynchronous alteration map (AAM) which describes the non-linear change. 2DCOR can then be used to understand the relationship between the chemical changes but 2DCOR can also resolve the sequence of the changes. A workflow for GC×GC-MS will be demonstrated. Special attention will be given to what differentiates the GC×GC work flow from GC for both the ALA and 2DCOR.

O-2

APPLICATION OF GC×GC FOR THE INVESTIGATION OF FERMENTED BEVERAGES

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Abstract

Fermentation is a byproduct of microbial metabolism. When beverages become fermented, the microorganisms involved produce volatile organic compounds (VOCs). It is important to examine the VOC profile of fermented beverages to understand their consumer perception and health considerations. Traditionally, VOCs are detected by gas chromatography-mass spectrometry (GC-MS). However, more complex matrices, like fermented products, benefit from the enhanced separation of comprehensive two-dimensional gas chromatography (GC×GC). This study aimed to develop an understanding of the VOCs related to different fermented beverages through a nontargeted lens via comprehensive two-dimensional gas chromatography with dual detection using flame ionization detection and time-of-flight mass spectrometry (GC×GC-FID/TOFMS). Data analysis focused on developing a complete VOC profile of fermented products. Moreover, analysis aimed to identify differentiating VOCs to understand differences between products and distinguish the drink matrix from the microbial matrix. Results indicate that it is possible to attribute VOCs to one component of a complex sample, creating a microbial VOC profile and a base VOC profile. It was possible to discern products from one another, including contaminated products from non-contaminated products. Data visualization consisted of Principal Component Analysis (PCA) scores and loadings plots, bar chats, Hierarchical Cluster Analysis (HCA), and Partial Least Squares Discriminant Analysis (PLS-DA). Data analysis software was capable of processing and aligning samples with high levels of biological variation, making GC×GC a promising analytical tool for market research and regulation. Future studies will aim to correlate VOCs detected via GC×GC-FID/TOFMS to the microbial genetics of fermented products.

O-3

EXPLORING PFAS IN CONSUMER GOODS USING TWO-DIMENSIONAL GAS CHROMATOGRAPHY AND HIGH-RESOLUTION MASS SPECTROMETRY

David E. Alonso, Joe Binkley

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Abstract

Enormous quantities of Polyfluoro- and perfluoroalkyl substances (PFAS) have been produced through telomerization and electrochemical fluorination since the late 1940s. PFAS are a group of anthropogenic chemicals produced to improve the physical and chemical characteristics of products such as food packaging, cookware, textiles, paints, flame retardants, and stain-resistant clothing. PFAS increase heat, water, and oil resistance in these consumer products. Humans can be exposed to these harmful substances through ingestion and inhalation. Therefore, it is crucial that researchers screen these toxic chemicals on a regular basis. Unfortunately, monitoring hazardous chemicals such as persistent organic pollutants (POPs) is challenging due to the complexity of sample matrices. The goal of this study was to develop an analytical methodology for the comprehensive screening of complex samples for volatile and semi-volatile PFAS, as well as additional emerging contaminants. The protocol included the application of effective sample introduction techniques, as well as the design of general data acquisition and processing strategies for quick analysis of a wide variety of consumer goods. Different ionization modes: 1) Electron Ionization (EI), 2) Positive Chemical Ionization (PCI), and 3) Negative Chemical Ionization (NCI). The rich GCxGC-HRTOFMS data were used to annotate different classes of PFAS and additional harmful chemicals.

O-4

AN ANALYSIS OF FRESH AND USED AIRCRAFT OIL: AN INDICATION OF EXPOSURE PATHWAY POSSIBILITY TO INORGANIC AND ORGANIC POLLUTANTS

Kevin Hayes, Gwen O'Sullivan

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Abstract

An elemental analysis was conducted on matched pairs of new and used aircraft engine oils for various aircraft engine types (piston, turboprop, and jet). The analysis aimed to determine what, if any, accumulation or loss of oil and fuel additives may occur with engine use. Losses in elemental loadings from new to used oils imply that there may be a possibility for the element-containing compounds to enter the pneumatic system of bleed air pressurized aircraft at a higher rate than that of oil attrition and potentially impact cabin air quality. Inductively Coupled Plasma Optical Emission Spectroscopy (ICP OES) was employed to complete the elemental analysis, and results describe a greater than twenty percent loss of phosphorus from new to used jet oils and a significant accumulation of lead in the oil of piston aircraft (Range: <LOD to 6821 ± 83 mg kg^{-1}; n=2).

In an effort to speciate and identify compounds of concern, the oil samples were introduced to a flow modulated GCxGC-TOFMS (SepSolv BenchTOF). Data was analyzed utilizing Chromspace and Analyzer Pro XD. The new and used oils were compared, and compounds exclusive to the used oils were screened to determine the possibility of increased risk associated with hazardous accumulations in the used oil. This used oil, which is adulterated by either amendment or contamination, is not fully described to individuals who may interact with the product, and therefore an apparent risk is potentially unmitigated.

O-5

SNIFF SMARTER: EMPOWERING GC–O WITH TRAP-BASED ENRICHMENT AND GC×GC FOR ADVANCED AROMA PROFILING

Lina Mikaliunaite[1], Khaled Murtada[2], Laura McGregor[3], Matthew Edwards[2], Jonathan Grandy[2], Lucy Hearn[4]

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Abstract

Gas chromatography–olfactometry (GC–O) integrates human sensory perception into analytical workflows by combining the nose with chromatographic instrumentation. While mass spectrometry provides molecular information, it cannot describe odour attributes, making GC–O essential for linking chemical composition to sensory perception.

Analysis is complicated by several factors. Many odorants occur at trace levels, with detection thresholds below the sensitivity of common detectors, creating a gap between measurable compounds and those perceived by the human nose. Additionally, co-elution in complex matrices, such as citrus fruits, can obscure identification of the compounds responsible for specific aromas.

Trap-based enrichment addresses these challenges by combining several sampling stages into a single run, focusing analytes on an electrically-cooled trap. This enhances sensitivity, allowing previously undetectable odorants to be confidently identified by mass spectrometry.

Comprehensive two-dimensional gas chromatography (GC×GC) further improves separation by resolving co-eluting compounds, producing cleaner spectra and enabling distinct assignment of compounds to sensory events.

This integrated workflow provides high-resolution aroma profiling, bridging analytical chemistry and sensory science, and is broadly applicable to flavour research, quality control, and product development in the food and beverage sector.

O-6

LEVERAGING LOCAL LIBRARIES FOR POSITIVELY PARSIMONIOUS PEAK TABLES

James Harynuk

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Abstract

GC×GC- MS shines when doing non-target analysis, and is an incredibly important tool for studying human health, human exposures to exogenous compounds, and transformations of compounds released into the environment. The enhanced separation and expanded separation space permits us to deliver very pure peaks to the mass spectrometer, free from many coelutions and also free from bleed from the primary column. This pure peak then results in a much cleaner, high quality mass spectrum, regardless of the mass spectrometer used. If a high resolution mass spectrometer is used, we then know the exact mass (and thus elemental composition) of every ion in the spectrum. This is a truly important tool for studying these problems. The inspiration for this work lies in the problem of what to do with unknown unknowns – those compounds not in any library? We have been exploring new, simple ways to improve the consistency in our reporting of compounds in non-target analyses. Using a combination of a user library and relatively simple tools, we can ensure that we always recognize the same compounds across various samples and sample types, even if we do not know exactly what the compound is. This should improve the quality of downstream data analysis, and potentially help standardize some of the tools used in GC×GC-MS data processing.

O-7

STRUCTURAL ELUCIDATION USING COMPREHENSIVE TWO-DIMENSIONAL GAS CHROMATOGRAPHY/TIME-OF-FLIGHT MASS SPECTROMETRY AND MACHINE LEARNING FOR UNKNOWN METABOLITES IN HELA CELLS

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Abstract

Gas chromatography–mass spectrometry (GC-MS) and comprehensive two-dimensional (GCxGC)-MS are a powerful tool for analyzing volatile organic compounds, widely used in both qualitative and quantitative applications. Compound annotation typically relies on comparison with commercial EI mass spectral databases, which are known for their reproducibility and instrument independence. However, many biologically relevant compounds remain unregistered, limiting the scope of metabolomic studies.

To address this challenge, we developed a deep learning model capable of predicting EI mass spectra from molecular structures. Using NIST 23 database for training of the prediction model, then approximately 100 million compound structures from PubChem and 75 million metabolite derivatives generated an AI-based EI mass spectral database for supporting metabolomics research. In this study, we focused on *N*-methyluridine monophosphate (*N*-methyl UMP), a metabolite derived from RNA modification, which is absent from existing databases.

We performed comprehensive analysis of water-soluble metabolites extracted from HeLa cells using GCxGC-TOFMS and characterized a TMS derivative of *N*-methyl UMP. The predicted spectra showed high concordance with experimental data, validating the model's accuracy. These results demonstrate that AI-based spectral prediction enables reliable identification of unknown metabolites, offering a promising approach for expanding the capabilities of GC-MS and GCxGC-MS in untargeted metabolomics.

[1] Lai, Z., Tsugawa, H., Wohlgemuth, G., Mehta, S., Mueller, M., Zheng, Y., Ogiwara, A., Meissen, J., Showalter, M., Takeuchi, K., Kind, T., Beal, P., Arita, M., and Fiehn, O. (2017) Identifying metabolites by integrating metabolome databases with mass spectrometry cheminformatics, Nat. Methods, 15(1):53–60.

O-8

TILE-BASED FISHER-RATIO ANALYSIS OF GC×GC-TOFMS DATA OF SPME SAMPLED VOCS PRODUCED FROM PSEUDOMONAS AERUGINOSA AND ASPERGILLUS FUMIGATUS

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Abstract

Pseudomonas aeruginosa and *Aspergillus fumigatus* are major pathogens found in the lungs of Cystic fibrosis patients. Their coexistence worsens lung function and leads to poor clinical outcomes. To investigate their metabolic interactions, we analyzed the volatile space of *P. aeruginosa* and *A. fumigatus* using SPME-GC×GC-TOFMS across four sample classes: Media, *P. aeruginosa* monoculture, *A. fumigatus* monoculture, and their co-culture. GC×GC-TOFMS provides high-resolution, high-sensitivity separation of complex metabolomic samples, but it also generates high-dimensional data that can be challenging to analyze. Therefore, feature selection and chemometric methods are essential to extract meaningful information. Additionally, peak table alignment issues due to retention time shifting across samples can in principle hinder the comparative analysis of multiple samples. Hence, we applied tile-based Fisher-ratio analysis using ChromaTOF Tile software to discover analytes that are statistically significant in concentration differences across samples with replicates while minimizing retention-time shifts. This platform generates a comprehensive hit list that links detected analytes across all samples. In summary, our study integrated tile-based analysis to generate a comprehensive peak table that relates all analytes in all samples in terms of up and down regulation of their concentrations. With further analyte identification, we have characterized analytes specific to each sample class to ultimately learn more about *Pseudomonas aeruginosa* and *Aspergillus fumigatus* pathogens and their co-culture.

O-9

IMPLEMENTING TILE-BASED FISHER RATIO ANALYSIS OF GC×GC-TOFMS DATA TO OBTAIN A MASTER PEAK TABLE OF ALL DETECTED ANALYTE COMPOUNDS IN MANY PETROLEUM-BASED SAMPLES

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Abstract

Historically, tile-based Fisher ratio (F-ratio) analysis of comprehensive two-dimensional gas chromatography time-of-flight mass spectrometry (GC×GC-TOFMS) data was developed for analysts to obtain a hit list to discover which analytes are top hits that most significantly distinguish the sample classes, with lower F-ratio hits being ignored and/or discarded. To broaden the scope of tile-based F-ratio analysis we explore the ability of the software to discover all analyte components that are detected in a set of samples, taking full advantage of the tiling aspect of the software which mitigates the adverse impact of sample run–to–sample run retention time misalignment. For this study a set of nine petroleum samples are simultaneously analyzed and statistically compared via p-testing to blank chromatograms to produce one comprehensive hit list. The pin locations and signal areas at the top m/z F-ratio are used together with replicate blanks to generate a master peak table (MPT) that in turn is used to generate sample-specific peak tables (SSPT) for each sample that are naturally retention-time aligned via the F-ratio software. The nine petroleum samples vary to a large extent in the identity and number of analytes present, and a large number of trace analytes were discovered across samples. This workflow also facilitated generating simulated distillation curves for the nine petroleum samples to provide further chemical distribution insight.

O-10

GcDUO: AUTOMATING GC×GC-MS DATA ANALYSIS VIA PARAFAC AND PARAFAC2

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Abstract

Multidimensional chromatography-mass spectrometry (MS) is a powerful analytical technique that integrates two or more chromatographic separations with MS, offering superior resolution, increased signal-to-noise, and selectivity for complex sample analysis. Despite its potential, its adoption remains limited due to data complexity and processing challenges. Chemometric approaches, particularly multiway models like Parallel Factor Analysis (PARAFAC), have proven effective in addressing these challenges by enabling the extraction of meaningful chemical information from multidimensional datasets. However, traditional PARAFAC is constrained by its assumption of data tri-linearity, which may not be valid in all cases, where data have misalignments. To overcome these limitations, we present GcDUO, an open-source data processing software that enables annotation, deconvolution, and analysis of batch GC×GC-MS data (Llambrich et al., Briefings in Bioinformatics 2025, doi: 10.1093/bib/bbaf080, https://github.com/mariallr/GcDuo). GcDUO, implemented in R, accepts non-vendor-specific standardized CDF files, and rearranges the data into four-dimensional tensor structures, preserving the GC×GC-MS data structure. GcDUO integrates advanced chemometric methods, including PARAFAC and PARAFAC2, for a more accurate and comprehensive analysis. PARAFAC is particularly useful for deconvoluting overlapping peaks and extracting pure chemical signals, while PARAFAC2 relaxes the tri-linearity constraint, allowing batch analysis for samples. GcDUO achieves both high-resolution peak detection and robust quantification across complex GC×GC-MS datasets. The software was validated against the gold-standard software for comprehensive GC, demonstrating a high correlation (R^2 = 0.9) in peak area measurements, confirming its effectiveness and reliability. GcDUO provides a valuable, open-source platform in the comprehensive chromatography field, enabling more accessible and customizable data analysis.

O-11

LEVERAGING 2D-LC TO IMPROVE METHOD UNDERSTANDING AND ROBUSTNESS FOR THERAPEUTIC BIOMOLECULES

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Abstract

Two-Dimensional Liquid Chromatography (2D-LC) has emerged as a powerful tool for the separation and analysis of complex mixtures in biopharmaceutical development. Common use cases for 2D-LC applied to the biopharmaceutical pipeline will be discussed, primarily focused on the heart-cutting analysis mode. These case studies include the MS identification of chromatographic peaks stemming from a non MS-compatible method in the first dimension, assessing the molecule in different states based on the selection of the 2D method conditions. Additionally, a peak purity assessment can be conducted to understand the potential presence of co-eluting species. Throughout the talk, demonstration of 2D-LC as a tool to improve our understanding and robustness of methods used in the QC environment will be highlighted, in addition to situations where a single chromatographic separation is inadequate.

O-12

LEVERAGING MECHANISTIC AND MACHINE LEARNING MODELS TO SIMPLIFY TWO-DIMENSIONAL LIQUID CHROMATOGRAPHY (2D-LC) METHOD DEVELOPMENT FOR PEAK PURITY ANALYSIS

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Abstract

Due to safety and quality considerations, regulatory requirements necessitate both achiral and chiral purity analysis of a small molecule human drug. For the final drug substances and drug products, this characterization process typically consists of two separate sets of method development efforts: one via achiral LC separation and one via chiral SFC or LC separation. Although such workflows are well established in analytical labs in the pharmaceutical industry, it can be a time-consuming process. In contrast, 2DLC can achieve both achiral and chiral peak purity assessment in a single shot and further increase automation and efficiency in method development. However, the usage of 2DLC systems has largely been limited due to the complexity in method development, partly associated with the number of parameters and settings involved. Particularly, unlike RPLC achiral separation in which chromatographic retention can be predicted with reasonable accuracy via certain theories such as LSS, it is well known that chiral separation is difficult to predict using retention models and requires screen of columns and mobile phases to arrive at an initial condition. Further optimization would require additional efforts. Our work demonstrated that 2DLC method development can be simplified and made more efficient through a combination of mechanistic and machine learning modeling.

O-13

TWO-DIMENSIONAL LIQUID CHROMATOGRAPHY ISOLATION AND QUANTIFICATION OF IMMUNOGLOBULIN G AND EXOSOMES FROM CELL CULTURE MEDIA USING CAPILLARY-CHANNELED POLYMER FIBER COLUMNS

Chris Topper, R. Kenneth Marcus

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Abstract

Extracellular vesicles (EVs) are small membrane-bound particles that are naturally released by cells into the extracellular environment. Exosomes constitute a subset of EVs with a characteristic size range of 30 – 150 nm. Exosomes are produced by nearly every cell type in the body and can be found in virtually all biological fluids. They share the same integral membrane proteins as their originating cell and thus can be used as biomarkers to identify and monitor disease.

Monoclonal antibodies (mAbs) such as IgG are primarily produced by culturing Chinese hamster ovary (CHO) cells and then passing the culture media through a Protein A (ProA) affinity chromatography column to isolate the antibodies. The waste effluent from this process, however, contains valuable EVs that are ultimately discarded. Therefore, the isolation of exosomes from CHO cell waste streams presents an opportunity for by-product valorization.

We describe a two-dimensional liquid chromatography platform employing columns packed with capillary-channeled polymer (C-CP) fibers to isolate both IgG and exosomes from CHO cell supernatant. The first dimension utilizes ProA to isolate and quantify IgG, while hydrophobic interaction chromatography (HIC) is used in the second dimension to isolate and quantify exosomes from the 1D effluent. Thus, we demonstrate a convenient framework for characterizing any CHO cell culture into both its IgG and exosome production traits, providing practical insights into the co-production of these two, disparate biotherapeutics, as well as a means for extracting valuable EVs during the production of mAbs, converting waste into dollars.

O-14

TWO-DIMENSIONAL LIQUID CHROMATOGRAPHY-MASS SPECTROMETRY APPLICATIONS IN PHARMACEUTICAL DEVELOPMENT

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Abstract

As the chemical complexity of pharmaceutical products increases, developing next generation analytical methodologies is essential to assess quality attributes of these complex therapeutic modalities. In this presentation, various implementations of two-dimensional liquid chromatography and mass spectrometry will be discussed that aim to improve the characterization of synthetic pharmaceutical molecules. Specifically, we will present advantages and applications of coupling different HPLC modes in a 2D-LC format, such as ion pairing reversed phase, hydrophilic interaction liquid chromatography, size exclusion chromatography, and anion exchange chromatography towards improved characterization of oligonucleotide and antibody-oligonucleotide conjugates.

O-15

DEVELOPMENT OF HARDWARE AND SOFTWARE APPROACHES TO COMPREHENSIVE CAPILLARY 2D-LC

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Abstract

Capillary liquid chromatography provides separations at much lower flow rates than standard analytical-scale liquid chromatography, greatly reducing the waste generated during analysis. Enhancement in MS signal for LC-MS can also be observed when low mobile phase flow rates are employed. Current 2D-LC separations are primarily performed with standard analytical-scale columns, thus requiring significantly greater flows that can lead to compatibility issues with MS detection. In this presentation, progress towards a comprehensive 2D-LC platform that uses capillary-scale columns in both dimensions is described. Method development has focused on a RP x RP separation of a standard small molecule test mixture and improvement of sampling rates by increasing the speed of the second dimension separation. Pump control software and data plotting software, developed in Java and Python respectively, will also be described.

O-16

NO MORE SPLIT ENDS? FLOW-MODULATED GCXGC-QMS ANALYSIS WITH OUT SPLITTING OFF THE GC FLOW

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Abstract

Flow-modulated two-dimensional gas chromatography (GCxGC) is an excellent solution for low-boiling point compounds that are difficult to separate by thermal modulation without cryogens. Pairing a quadrupole mass spectrometer (QMS) with GCxGC can offer better dynamic range, increased sensitivity, and lower cost to GCxGC applications, however, many MS instruments can not accept the high flow rate coming from flow-modulated GCxGC.

In this study, a flow modulator was installed in a QMS system designed to accept high GC flow rates, and tested using perfume and pesticide applications. Results detailing the sensitivity and effectiveness of the QMS system with high GCxGC flow input will be discussed, as well as a comparison to thermal modulation results from the same system.

O-17

ONE SHOT TENSOR DECOMPOSITION OF FULL-SCALE TWO-DIMENSIONAL GAS CHROMATOGRAPHY DATASETS FOR RESOLVING PETROCHEMICAL GROUPS

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Abstract

Tensor decomposition methods such as PARAFAC and PARAFAC2 have been proposed for resolving co-elutions, and extracting purified spectra in two-dimensional chromatography datasets. However, the application of these methods is usually restricted to local regions-of-interest (ROIs). Therefore, additional pre-processing steps, are required and processing multiple samples at once can be difficult due to retention time shifts (e.g., it is difficult to define a ROI that captures the same analyse signal in multiple samples).

This work presents an application of fast, shift-invariant tensor decomposition, which was used for the decomposition of full-scale GCxGC-VUV datasets. The results show that chemical groups can be resolved, which are non-separable with conventional, template-based approaches, such as aliphates and olefines, Furthermore, the extracted peak areas are in good agreement with the quantitative results of the reference methods (e.g., bromine number for olefines).

O-18

TWO-DIMENSIONAL GAS CHROMATOGRAPHY/ELECTRON AND CHEMICAL IONIZATION HIGH-RESOLUTION MASS SPECTROMETRY FOR CHEMICAL CHARACTERIZATION OF COMPLEX MIXTURES

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Abstract

Two-dimensional gas chromatography is a powerful technique for the separation of organic compounds in complex mixtures. However, even the exceptionally high resolution of this technique is not high enough to resolve all compounds in many complex mixtures, such as aviation fuels. Therefore, this method is often coupled to mass spectrometry detection. However, the commonly used electron ionization (EI) mass spectrometry method often causes so extensive fragmentation that no stable molecular ion is generated, which hinders or prevents compound identification as no information is obtained about the MW of the analytes. This issue can be addressed by using positive-ion mode chemical ionization (CI) with methane reagent gas as this approach involves a much lower-energy ionization process. CI with methane gas usually yields ions that contain the intact analyte molecule (protonated molecule or its fragment ion formed by elimination of a hydrogen molecule) and hence provides MW information for the analytes. This is true even for large saturated hydrocarbons and long-chain aliphatic alcohols. Furthermore, CI generates structurally informative fragment ions that complement the more limited structural information obtained from EI mass spectra.

O-19

EFFECTIVE (AND MULTIDIMENSIONAL) STRATEGIES FOR THE CAPTURE AND SEPARATION OF VOLATILE PFAS IN AQUEOUS AND GAS PHASE

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Abstract

Per- and polyfluoroalkyl substances (PFAS) are environmentally persistent and toxic synthetic compounds. Volatile and neutral PFAS, used in products such as firefighting foams and non-stick coatings, act as precursors to persistent acids like PFOS and PFOA, making their accurate monitoring essential. While analytical methods for legacy ionic PFAS in water are well established, there is a critical gap in efficient strategies for neutral, volatile PFAS. The main challenge consists in their effective capture and preconcentration from the gas phase and their efficient introduction into the analytical instrument. The complexity of real-world samples further complicates analysis by introducing matrix interferences that can mask the presence of these low-level contaminants. This work highlights how Solid-Phase Microextraction (SPME), coupled with comprehensive two-dimensional gas chromatography (GC×GC) and time-of-flight mass spectrometry (ToF/MS), provides a powerful platform for volatile PFAS analysis in both air and water. The workflow enables highly sensitive quantitation at sub-part-per-billion levels, advancing environmental monitoring and risk assessment. SPME Arrow devices with diverse sorbent chemistries were evaluated to compare preconcentration efficiencies. The optimized method was applied to monitor emissions from paint samples, revealing the presence of several classes of fluorinated compounds. For such complex mixtures, the orthogonal separation capacity of GC×GC proved indispensable, resolving co-eluting peaks and enabling confident analyte identification. This workflow enhances both preconcentration efficiency and molecular specificity, enabling reliable quantitation of volatile PFAS alongside the discovery of previously unrecognized species. Together, these advances provide a critical foundation for addressing current gaps in PFAS monitoring strategies.

O-20

OPTIMIZATION OF DIRECT THERMAL EXTRACTION PARAMETERS FOR ANALYSIS OF HIGH-WATER CONTENT SAMPLES USING COMPREHENSIVE TWO-DIMENSIONAL GAS CHROMATOGRAPHY

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Abstract

The characterization of volatile organic compounds (VOCs) associated with abiotic samples can be complex due to the varied composition of sample matrices. For instance, concrete is composed of different types of cement, aggregates, and additives mixed in varying ratios with water, impacting subsequent sampling methodology and analyses. We analyzed the VOCs of concrete cores via direct thermal extraction (DTE), using a LECO Pegasus 4D GC×GC–TOFMS equipped with a Gerstel MPS thermal desorption unit, and observed significant shifting in the first-dimension retention times (RTs) of VOCs with lower boiling points. This was attributed to the release of cementitious hydrates into the sample headspace at high temperatures (> 80°C) during DTE that were condensing as water or ice plugs in the cooled injection system (CIS). Here, we aimed to optimize DTE parameters to stabilize first-dimension RTs for highly volatile compounds while maximizing peak intensities. We utilized central composite design (CCD) to determine the optimal initial temperature and secondary hold time of the cooled injection system (CIS) for water elimination and volatile trapping onto the Tenax® TA CIS liner. Analytical standard mixes in water and concrete samples were tested in the CCD. We observed stable first-dimension RTs at initial CIS temperatures ≥ 30°C and secondary hold times in the range of 5-20 min, while the relative abundance of individual VOCs remained largely unaffected. Utilization of the optimized DTE parameters to analyze concrete samples yielded over 350 chromatographic peaks and demonstrated stable first-dimension RTs.

O-21

SELECTION, OPTIMIZATION, AND VALIDATION OF THERMAL DESORPTION FOR ANALYSIS OF VOCS AND PAHS IN COMBUSTION SMOKE

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Abstract

Smoke from fires releases large quantities of hazardous chemicals into the atmosphere causing poor air quality at local, regional, and global scales. Due to the complexity of smoke emissions, the health impacts from inhalation and dermal exposure vary with fuel type and combustion conditions. Accurate emissions characterization data is critical for understanding health risks associated with smoke exposure. To date, smoke characterization has been predominately conducted utilizing filter extracts and standard GC-MS. This method has several limitations including lengthy extraction times, excessive use of toxic solvents, and cluttered chromatograms resulting in peak coelution. The use of thermal desorption (TD) combined with two-dimensional gas chromatography offers an appealing alternative to traditional methods by improving recovery, eliminating solvent use, and enhancing chromatographic clarity through second dimension separation. However, limited work has been published optimizing these techniques for smoke emissions.

This research sought to optimize sorbent bed selection and TD method parameters to allow non targeted analysis of smoke emissions. We explored the application of 6 unique sorbent bed combinations to optimize analyte retention of smoke compounds with varying polarity and volatility. Following sorbent selection, a design of experiment approach was applied to optimize the analytical desorption parameters (time, temperature, and flow) and conditioning methodology (time, temperature). The method was validated using smoke collected from lab-scale combustion chamber under controlled conditions. This study establishes a strong analytical foundation to improve our understanding of both primary and secondary smoke emissions, supporting more accurate risk assessment and informing public health and environmental policy.

O-22

ON-CHIP ELECTROMEMBRANE SURROUNDED SOLID-PHASE EXTRACTION COUPLED WITH GC-MS FOR DETERMINATION OF DRUGS IN BIOLOGICAL FLUIDS: A GREEN AND SENSETIVE TECHNIQUE

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Abstract

Electromembrane extraction (EME) is a promising sample preparation technique with high selectivity, low solvent use, and preconcentration of charged analytes. However, EME often suffers from low recovery, and instability of the supported liquid membrane (SLM). Coupling EME with SPME enables selective ion transfer and cleanup, while SPME provides strong enrichment factor and instrument compatibility.

Here, a microfluidic electromembrane-surrounded solid-phase microextraction (EM-SPME) method was developed for antidepressant determination in complex biological matrices. The EME system comprised donor and acceptor phases separated by a porous membrane containing the SLM. A poly(3,4-ethylenedioxythiophene)–graphene oxide (PEDOT–GO) nanocomposite was electrodeposited onto the SPME fiber, serving as both acceptor electrode and extraction medium. Under an electric field, analytes migrated across the SLM and were absorbed onto the fiber, then thermally desorbed in the GC–MS inlet for detection.

Six tricyclic antidepressants—amitriptyline, nortriptyline, imipramine, desipramine, maprotiline, and sertraline—were targeted. Optimized conditions yielded detection limits of 0.005–0.025 μg L^{-1}. The method showed excellent linearity: 0.010–500 μg L^{-1} for imipramine and sertraline, 0.025–500 μg L^{-1} for amitriptyline, nortriptyline, and desipramine, and 1.000–250 μg L^{-1} for maprotiline.

The technique was applied to human bone marrow aspirate, urine, and plasma, with recoveries of 93–105%, confirming reproducibility and applicability. Compared with conventional SPME, EM-SPME reduced carryover and matrix effects while maintaining high sensitivity. This work introduces a versatile, miniaturized platform for trace drug analysis that combines microfluidics and two extraction techniques, suited for bioanalytical applications in complex matrices.

O-23

IDENTIFYING THE TRANSITION FROM ANTE-MORTEM TO POST-MORTEM ODOR IN CADAVERS IN AN OUTDOOR ENVIRONMENT

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Abstract

This study investigates the transition from ante-mortem to post-mortem odor in human remains during the early post-mortem period in an outdoor environment. Three cadavers (donors) were placed at an outdoor human decomposition facility, and volatile organic compounds (VOCs) were collected and analyzed using thermal desorption coupled with comprehensive two-dimensional gas chromatography-time-of-flight mass spectrometry (TD-GC × GC-TOFMS). The key findings revealed that nitrogen-containing compounds were predominant in early post-mortem VOC profiles, driven by enzymatic and bacterial activity. Esters, alcohols, and halogenated compounds were also identified, with esters linked to microbial transformation and alcohols possibly formed by lipid peroxidation. Ante-mortem VOCs were persistent across samples, influenced by skin microbiota and environmental factors like UV radiation, complicating the detection of decomposition odor. Post-mortem VOCs became more prominent after ADD 73.4(experimental day 3), signaling the transition to the bloat stage of decomposition. Variations in sample collection methods and external factors such as temperature were found to affect VOC abundances. This study provides critical insights into odor transition and has implications for the use of search and rescue (SAR) and human remains detection (HRD) dogs. Further research is needed to standardize methods and assess odor transitions across diverse environments and seasons.

O-24

USING SCANNING ELECTRON MICROSCOPY WITH ENERGY DISPERSIVE X-RAY SPECTROSCOPY AND COMPREHENSIVE TWO-DIMENSIONAL GAS CHROMATOGRAPHY TO DEVELOP A COMBINED APPROACH TO GREEN GUNSHOT RESIDUE ANALYSIS IN THE FORENSIC LABORATORY

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Abstract

Gunshot residue (GSR) is expelled as a "plume" of vaporized material during a firearm discharge event. As it disperses, condenses, and settles on surfaces in vicinity of the discharge, it creates chemical depositions that provide essential information for forensic casework involving criminal firearm usage. GSR contains organic (OGSR) and inorganic (IGSR) components, both of which may carry evidentiary value. Traditional analyses involve the targeted point analysis of IGSR via scanning electron microscopy - energy dispersive x-ray spectroscopy (SEM-EDS), and conclusions are drawn based on a well-established elemental and morphological profile "characteristic" to IGSR. This method has lost impact as a standard for forensic analysis due to increased use of "green" - heavy metal-free - ammunition, which omits the lead, barium, and antimony that are traditionally seen as the "characteristic" markers of GSR. This study aimed to develop a holistic approach to the analysis of GSR using both SEM-EDS as a targeted approach to IGSR analysis and comprehensive two-dimensional gas chromatography – time-of-flight mass spectrometry (GC×GC-TOFMS) as a nontargeted approach to OGSR analysis. GSR samples were collected from the hands of shooters at a police firearms recertification training using the tape lift method. SEM-EDS was used to identify IGSR and develop new morphological and elemental profiles for heavy metal-free residues. OGSR components have been positively identified through sample analysis via liquid extraction, chromatographic separation via GC×GC-TOFMS, and data processing. Future work on this project will include the optimization of data processing methods and the development of SPME as an alternative sample extraction method.

O-25

UNEXPECTED SOLVENT SELECTIVITY EFFECTS ENCOUNTERED IN TWO-DIMENSIONAL LIQUID CHROMATOGRAPHY SEPARATIONS OF NON-IONIC COPOLYMER SURFACTANTS

Ella Sontowski[1], Tina Dahlseid[1], Dwight Stoll[2], Kristeen Joseph[3], Peilin Yang[3]

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Abstract

Analysis of non-ionic copolymer surfactants is challenging due to complexities of the mixture of molecules that results from industrial-scale synthesis involving heterogeneous starting materials. Two-dimensional liquid chromatography (2D-LC) involving hydrophilic interaction (HILIC) and reversed-phase (RP) separation modes has been shown to be particularly effective for this purpose. The HILIC mode is highly selective for the separation of oligomers that vary in the number of hydrophilic (e.g., ethylene oxide [EO]) repeating units, and the RP mode is highly selective for the number of hydrophophic repeating units, such as polypropylene oxide (PO). In our work on separation of EO/PO copolymer mixtures by 2D-LC we have discovered that certain mobile phase compositions used for the HILIC separation yield unexpected selectivities for isomers of these copolymers that can result from differences in the way the EO/PO oligomers are assembled during the synthesis reaction. In this presentation we will share results of a systematic study of mobile phase constituents that exhibit these selectivity effects, and demonstrate the utility of these new-found effects in ultra-high resolution 2D-LC separations of EO/PO copolymers showing that they provide insights into the species-level composition of these complex mixtures that was not possible previously.

O-26

COUNTING DOUBLE BONDS: GC×GC–FID FOR PLASTIC PYROLYSIS OILS

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Abstract

Plastic pyrolysis oils (PPOs) are unresolved complex mixtures in which the level and speciation of unsaturation govern storage stability, upgrading severity, and the quality of fuel-range cuts. Yet routine, transferable olefin quantitation is hampered by extensive coelution and isomer diversity. This talk presents a GC×GC–FID workflow that “counts double bonds” by selectively derivatizing olefins to reposition them within the two-dimensional separation space, cleanly separating them from paraffins, naphthenes, and aromatics. The method produces carbon-number- and class-resolved olefin distributions (wt.%) for whole oils and distillates in the gasoline, jet, and diesel distillation range.

P-1

TEMPERATURE-OPTIMIZED POROUS GRAPHITIC CARBON CHROMATOGRAPHY FOR IMPROVED RESOLUTION OF HIGH-MANNOSE GLYCANS

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Abstract

High-mannose N-glycans are critical biomarkers in glycobiology and disease research, but their separation remains analytically challenging due to subtle structural differences among glycoforms. In this work, we systematically evaluate the effect of column temperature (50–110 °C) on the porous graphitic carbon (PGC) separation of procainamide-derivatized RNase B glycans (Man_5–Man_9). Quantitative metrics including peak resolution, theoretical plates, peak symmetry, and method precision were assessed across temperature ranges. Our results reveal that moderate temperatures provide the best balance of resolution and reproducibility, with significant improvements in glycoform separation. For example, resolution between Man_8/Man_7 increased from 1.00 at 50 °C to 1.27, and Man_7/Man_6 from 1.80 to 2.66. However, excessive heating (≥100 °C) reduced separation quality due to peak broadening. These findings highlight the strong influence of thermal conditions on PGC retention mechanisms and demonstrate how temperature optimization can enhance multidimensional chromatography workflows for glycans. By providing practical temperature guidelines, this study supports more robust and reproducible glycoform analysis, enabling improved biomarker discovery and glycomics applications.

P-2

NON TARGET ANALYSIS OF WASTE PLASTIC PYROLYSIS OILS (WPPO) BY GCXGC-HRTOFMS

Christina Kelly, Joseph E Binkley

LECO Corporation, St. Joseph, MI, USA

Abstract

Waste plastic pyrolysis oils (WPPO) are of growing interest as a source of more environmentally friendly alternative feedstock for producing chemicals and fuels. However, as WPPO are often produced from diverse sources with varying degrees of purity and cleanliness, more comprehensive analysis becomes necessary as compounds that are not typically found in traditional petrochemical sources can be present in these oils. Targeted screening is not enough to fully safeguard processes from potentially undesirable contaminants, which can reduce efficiency of reactions and foul production lines. To fully understand the chemical composition of such complex mixtures, nontargeted analysis is essential. This presentation focuses on analysis of WPPO using an unparalleled nontarget discovery tool: comprehensive two-dimensional gas-chromatography coupled to high-resolution time-of-flight mass spectrometry (GCxGC-HRTOFMS) capable of multi-mode ionization with electron ionization (EI), positive chemical ionization (PCI), and electron-capture negative chemical ionization (ECNI). This multidimensional analysis provides not only the enhanced chromatographic resolution of GCxGC, which separates individual oil components chromatographically in an easy-to-comprehend layout of fairways of similar chemical structures, but also the powerful analyte identification abilities of complementary ionization modes that can provide both detailed structural information and the high mass-accuracy molecular formulae for individual species.

P-3

A NEW SOFTWARE TOOL FOR STANDARDIZATION OF GCXGC GROUP-TYPE TEMPLATES

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Abstract

As routine methods using comprehensive multidimensional gas chromatography (GCxGC) gain acceptance, creative solutions to traditional standardization challenges have emerged. One such solution is the LECO ChromaTOF feature “Classification Correction,” which allows for the simple adjustment of group-type templates between samples collected with different acquisition parameters. Designed to compensate for retention time shifts due to routine GC maintenance such as column trimming or replacement, “Classification Correction” enables the creation of shareable methods from system-to-system and provides valuable time-savings, easing the adoption of newer, more efficient routine GCxGC analyses. This poster shows the utility of the ChromaTOF software package for not only the bulk group-type analysis of alternative aviation fuels, but also the power of detailed hydrocarbon analysis made possible when GCxGC is coupled to time-of-flight mass spectrometry (TOFMS)

P-4

DEVELOPING 2D MZCOMPARE FOR SINGLE COMPREHENSIVE TWO-DIMENSIONAL CHROMATOGRAPHY TIME-OF-FLIGHT MASS SPECTROMETRY CHROMATOGRAMS: SUBSTANTIAL RESOLUTION ENHANCEMENT IN THE CONTEXT OF STATISTICAL OVERLAP THEORY

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Abstract

Accurate identification of all detectable components in a single comprehensive two-dimensional (2D) gas chromatography time-of-flight mass spectrometry (GC×GC-TOFMS) chromatogram is fundamentally of interest in the field. There are commercial software tools intended for this purpose, which is to provide a peak table listing analyte retention times on both dimensions and corresponding compound name for identification based upon matching a given analyte peak to a library of mass spectra. However, the performance accuracy for these commercial peak table generating software tools is not fully validated. Our current research is to explore this issue, and to provide a new algorithmic software approach called 2D mzCompare to generate optimally accurate peak tables for GC×GC-TOFMS. For this purpose, we simulate realistic GC×GC-TOFMS data so the exact number and identification of analytes is known *a priori*. Utilizing an in-house library containing many similar compounds, the simulated chromatograms vary in complexity, so we can apply our new software to identify and verify the number of compounds present under numerous application scenarios. In summary, our study presents a new software approach to provide a validated number of analytes in a given peak table and discover the limit of correctly identifying components relative to the chromatogram complexity for both simulated and real GC×GC-TOFMS data in the context of the statistical overlap theory (SOT) which is used to define the level of 2D chromatographic saturation.

P-5

GC×GC FOR THE MODERN LABORATORY: ENHANCING AROMA PROFILING AND PRODUCT COMPARISON

Lina Mikaliunaite[1], Laura McGregor[2], Khaled Murtada[3], Matthew Edwards[3], Jonathan Grandy[3], James Ogden[2]

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Abstract

The aroma profiles of herbs, spices, and essential oils play a key role in food and fragrance products, making it essential for manufacturers to measure them in order to assess product composition, detect odour taints, and maintain quality control.

Comprehensive two-dimensional gas chromatography (or GC×GC) has been proven to provide improved separation of such complex mixtures, by coupling two distinct separation mechanisms (e.g. by volatility and then polarity). With GCxGC, we know we can gain greater insight into sample composition, but the challenge then becomes "*what am I going to do with all of this data?*". Unfortunately, this has led to GC×GC being less attractive for GC–MS users who could truly benefit from the extra separation capacity.

Here, we demonstrate a workflow that solves this challenge through automated pairwise comparison of the raw data, allowing reporting of key differences to be completed in a matter of minutes.

This approach uses probability-based subtraction to generate new 'subtracted' chromatograms that highlight the components increased in the sample versus the reference, and vice versa. This allows analysts to easily visualise key differences within a familiar user interface, while also making it simple to explain the results to non-chemists and other stakeholders. Importantly, the new chromatograms can be processed in the regular manner, for automated integration, library-searching and reporting of the key differences without the need for complicated statistical workflows.

We demonstrate the application of this approach for the pairwise comparison of curry powders and essential oils of different quality.

P-6

ENHANCING TD–GC×GC–TOF MS WORKFLOWS FOR THE RELIABLE IDENTIFICATION OF MALODOURS IN RECYCLED PLASTICS

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Abstract

The increasing focus on a circular economy has led to greater use of postconsumer recycled (PCR) plastics, especially in food and beverage packaging. This shift demands more rigorous quality control to ensure PCR plastics do not emit harmful compounds or unpleasant odours. However, conventional methods for odour detection, such as sensory panels, electronic noses and GC–MS, face limitations in sensitivity, specificity and efficiency.

These issues can be dealt with by combining thermal desorption with comprehensive two-dimensional gas chromatography and time-of-flight mass spectrometry (TD–GC×GC–TOF MS). While this workflow improves sensitivity, separation and detection of the odour profiles, efficiently identifying the compounds responsible for malodours remains a challenge.

In this poster, we introduce a workflow that overcomes this limitation by using probability-based subtraction of the raw data, allowing reporting of key differences to be completed in a matter of minutes. In simple subtraction, small relative differences between high-intensity peaks (such as aliphatics from polymers) often dominate the subtracted chromatogram. This new approach suppresses these small relative differences, resulting in a clean background for easier identification of the compounds responsible for malodours.

Furthermore, the new subtracted datafiles can be processed in the usual manner, for automated integration, library-searching and fast reporting of the key differences. This straightforward workflow makes it easy to present results through a familiar user interface, without the need for advanced statistical knowledge.

P-7

ADVANCES IN METHOD DEVELOPMENT STRATEGIES AND TOOLS FOR TWO-DIMENSIONAL LIQUID CHROMATOGRAPHY

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Abstract

Two-dimensional liquid chromatography (2D-LC) is recognized as a versatile and powerful tool applicable to diverse analytical challenges ranging from resolution of molecules with multiple chiral centers to complex mixtures of biomolecules and industrial materials. However, a major barrier that prevents more users from realizing these capabilities in practice is the paucity of easy-to-use strategies and tools to support development of 2D-LC methods. In this presentation I will provide an update on several facets of our effort to address this need. This will include: 1) the development of a large, freely available database of retention data to support development of modeling and simulation tools; 2) the development of a freely available web-based simulator for 2D-LC that leverages our retention database; and 3) development of generic method development strategies that simplify decision-making during the 2D-LC method development process. I will also briefly touch on the role of machine learning in these efforts. Our aim with all these activities is to enable a more systematic approach to 2D-LC method development, so that we can lessen our reliance on trial-and-error experimentation and user experience compared to what has been done in the past. As part of my update, I will discuss their use in development of methods for applications ranging from small molecules to therapeutic biomolecules, and industrial surfactants. We expect that the proliferation of easy-to-use method development tools and strategies will both expand the application space for contemporary 2D-LC, and increase the number of users engaged in development of 2D-LC methods.

P-8

IDENTIFYING NON-BIOLOGICAL VARIANCE IN UNTARGETED ANALYSIS IN BREATH VOCS

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Abstract

Untargeted breath metabolomics offers a powerful avenue for biomarker discovery, enabling the detection of disease states, microbial interactions, and environmental exposures. However, these workflows are highly vulnerable to non-biological variance introduced during sample collection, handling, and analysis, which can obscure meaningful biological signals. This study aimed to identify and quantify such variance in untargeted volatile metabolomics.

Breath samples were collected from 67 participants, each of whom gave samples over a span of 5–6 weeks using Tenax® GR thermal desorption tubes (TDTs). For this study, participants' diets were controlled for 24 h prior to collection through the provision of boxed meals to regularize samples across participants and weeks. Samples were analyzed with comprehensive two-dimensional gas chromatography coupled to time-of-flight mass spectrometry (GC×GC–TOFMS; Leco Pegasus 4D). Most were analyzed one week post-collection, with a subset collected and analyzed as technical duplicates. Pairwise cosine dissimilarities between intra-subject and inter-subject samples were calculated across collection and analysis intervals.

Results showed that intra-subject breath profiles became increasingly dissimilar when samples were collected farther apart in time, indicating temporal drift. Inter-subject profiles displayed a similar trend, suggesting this drift was dominated by non-biological variance. Instrumental drift was identified as the largest contributor to variance across one to eleven weeks of analysis.

These findings demonstrate that non-biological sources, particularly instrument drift, can significantly impact untargeted metabolomics. By revealing consistent, time-dependent patterns of dissimilarity, this study highlights the importance of identifying, quantifying, and mitigating non-biological variance to support reliable biomarker discovery and biological interpretation.

P-9

ANALYSIS OF AROMA COMPOUNDS IN SPICES BY COMPREHENSIVE TWO-DIMENSIONAL GAS CHROMATOGRAPHY/TIME-OF-FLIGHT MASS SPECTROMETRY WITH MACHINE LEARNING-BASED STRUCTURE ELUCIDATION AND MOLECULAR FORMULA ESTIMATION

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Abstract

Since aroma components in food comprise numerous compounds, comprehensive two-dimensional gas chromatography–mass spectrometry (GCxGC-MS) is an effective technique. In GCxGC-MS, compound identification is commonly performed by searching mass spectral databases using electron ionization (EI). However, many compounds are often not registered in these databases. These require manual structural analysis, which can be difficult. To address the difficulty of manual structural analysis, we developed a structural elucidation method using machine learning-based mass spectral prediction. The predicted mass spectra can be used as a database, enabling structural estimation in the same way as conventional database searching. Furthermore, in this method, molecular formulas obtained from soft ionization (SI) data are used to narrow down candidate structures. However, a limitation was that multiple molecular formula candidates often remained. To overcome this, we also developed a technique to rank candidate formulas using a machine learning model. In this study, we report an application of the above method to the analysis of aroma compounds in spices by SPME-GCxGC-TOFMS.

In total, 518 compounds were detected by GCxGC-TOFMS. Integrated analysis using both EI and SI data enabled reliable identification of key aroma constituents of cardamom, including monoterpenes, sesquiterpenes, and furans. Compound annotation was based on combined evidence from NIST database searching, retention index, molecular ion and isotope information from SI, and accurate fragment masses from EI. When multiple candidate formulas were obtained, our machine learning model ranked them, allowing selection of the most plausible result. Even for database-unregistered compounds, molecular formulas and tentative structures could be estimated.

P-10

EXPERIMENTAL DEGRADATION AND GC×GC-TOFMS TO INVESTIGATE NEANDERTHAL TAR PRODUCTION METHODS

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Abstract

The identification of prehistoric adhesives on stone tools is crucial for understanding tool manufacture and use. At present, such adhesives are commonly analyzed using GC–MS, a method that requires solvent extraction and chemical derivatization of residues. These derivatized extracts are typically examined using targeted analytical workflows, which can limit interpretation of the broader technological and functional context of tool production and use. Prehistoric adhesives encompass a wide range of materials (e.g., plant resins, animal glues, gums), creating a need for sensitive, and broadly applicable identification approaches.

This study evaluates key methodological assumptions by investigating how controlled degradation affects the chemical signatures used to identify birch tar. Experimental birch tar was produced using both oxygen-poor (raised structures) and oxygen-rich (condensation, cobble-groove) methods, then subjected to ultraviolet irradiation and climate-chamber aging to simulate preservation processes. GC×GC–TOFMS revealed statistically significant differences among fresh samples, primarily driven by combustion-related compounds associated with oxygen-rich production methods. However, controlled degradation systematically altered these markers. UV irradiation preferentially reduced polycyclic aromatic hydrocarbons and monosaccharides that distinguish oxygen-rich production, while biomarkers used for birch tar identification (betulin, lupeol, betulinic acid) remained stable.

These results suggest that the absence of production-related markers in archaeological residues may reflect post-depositional alteration rather than specific manufacturing choices. The study highlights limitations of current analytical practices, including reliance on subjective peak annotation and the lack of comprehensive reference databases. Together, these findings underscore the need for continued methodological development and caution when using chemical evidence to reconstruct prehistoric adhesive technologies.

P-11

WHAT DO WE DO WITH ALL THAT DATA? COMPLEMENTARY DATA PROCESSING METHODS FOR TWO-DIMENSIONAL GAS CHROMATOGRAPHY AND MASS SPECTROMETRY.

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Abstract

Comprehensive two-dimensional gas chromatography combined with mass spectrometry generates a great deal of information. The different data analysis packages offer complementary features with different strengths, depending on the questions to be answered. A combined approach using different software systems is often needed to obtain a comprehensive view of the sample composition.

The first step in data analysis is often determination of the types of compounds that are present. Although this is traditionally done by database searching, it is helpful to make use of other data, such as accurate mass measurements and retention index matching. For samples that have repeating units such as petrochemicals, polyhalogenated compounds, and polymers, soft ionization such as field desorption or photoionization is helpful, with or without chromatographic separation. In fact, measurements made without chromatographic separation can reveal compounds that are not suitable for gas chromatography. It's important to know what we're missing! Modified Kendrick Mass Defect plots can provide an overview of the compound classes present, and that information is helpful to guide the examination of the GCxGC-MS data with dedicated GCxGC software and identify the regions on the 2D chromatogram where different compound classes may be found. Chemometric analysis such as offered with SpectralWorks' AnalyzerPro XD software is a powerful approach to identifying differences between samples.

Lastly, an automated method to determine the modulation period from the unprocessed one-dimensional chromatogram will be described with application to GCxCC-MS data from high-resolution and low-resolution mass spectrometers.

P-12

CHARACTERIZING PQSE'S ENZYMATIC ACTIVITY IN *PSEUDOMONAS AERUGINOSA* BY VOLATILE ORGANIC COMPOUND ANALYSIS WITH GC×GC-TOFMS

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Abstract

Pseudomonas aeruginosa is an opportunistic pathogen commonly associated with severe respiratory infections in immunocompromised patients. The pathogenic behaviors of *P. aeruginosa* are controlled by a cell-to-cell communication system called quorum sensing (QS). Many of the genes activated by QS include those responsible for forming biofilms and producing virulence factors. In *P. aeruginosa*, RhlR acts as a receptor and transcription factor for the Rhl QS system. Interestingly, several of the virulence factors produced by *P. aeruginosa* are regulated by an interaction between RhlR and a hydrolase, PqsE. Thus, the PqsE-RhlR interaction has become a target for antibiotic development and is a subject in need of further characterization. By using the increased peak capacity and superb sensitivity of comprehensive two-dimensional gas chromatography paired with time-of-flight mass spectrometry (GC×GC-TOFMS), it is possible to discover volatile substrates and products of PqsE enzyme activity. Previous work has utilized multiple *in vitro* assays to test small molecule binding in the active site of PqsE and inhibition of its esterase activity. However, to date, few *in vivo* assessments of PqsE catalytic activity exist. With GC×GC-TOFMS, the volatile organic compound (VOC), acetophenone, was discovered and utilized to measure dose-dependent inhibition of PqsE enzymatic activity *in vivo* by the small molecule inhibitor, Vorinostat. Furthermore, additional VOCs were identified that could add additional insight to the function of PqsE. Lastly, these findings can be used to create a targeted GC×GC method of screening potential inhibitors for their activity in *P. aeruginosa.*

P-13

A SUSTAINABLE APPROACH TO NONTARGETED ANALYSIS USING HYDROGEN AS A CARRIER GAS FOR GC×GC

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Abstract

Nontargeted screening can support the comprehensive characterization of true unknowns more successfully than targeted methods for one or several analytes. Not only does it provide a more complete picture of the sample, but it is also inherently greener than targeted methods. Instead of having to rerun samples multiple times to find different target analytes, a nontargeted approach provides the research with all the information that can be obtained from the sample in one run. Shorter run times are more efficient and greener. One nontargeted technique is comprehensive two-dimensional gas chromatography (GC×GC) with time-of-flight mass spectrometry (TOFMS), a separation technique that separates analytes based on two independent retention mechanisms. This study aimed to demonstrate the effectiveness, sustainability, and efficiency of hydrogen as a carrier gas for GC×GC-TOFMS. Using hydrogen over helium as a carrier gas leads to significantly shorter run times and can reduce carrier gas costs, both benefits that were observed in this study. Significant improvements in resolution were also seen for GC×GC-TOFMS analysis of forensic samples. Nontargeted analysis with GC×GC allowed for more peak capacity for unknown analytes instead of only targeted analytes in a specific range. With sustainable chemistry becoming more of a focus across the industry, the use of helium is less favorable, and alternative options are being sought. Acknowledging these enhancements when using hydrogen, this study demonstrated the effectiveness of hydrogen as a carrier gas through green chemistry metrics.

P-14

OPTIMIZING SOLVENT SELECTION AND DATA REDUCTION WORKFLOWS FOR GC×GC FIRE DEBRIS ANALYSIS

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Abstract

The use of comprehensive two-dimensional gas chromatography (GC×GC) is a newer technique for fire debris analysis. Offering improved resolution of complex mixtures compared to traditional one-dimensional gas chromatography (GC). Interpreting GC×GC data can be challenging and strenuous due to its high dimensionality and resolution. In this study, the influence of five solvents on the extraction of ignitable liquid residue (ILR) was evaluated. Chromatographic data from ILR samples were processed and analyzed using ChromaTOF Tile to build a working list of compounds for further chemometric analysis.

Chemometric analysis was conducted using Uniform Manifold Approximation and Projection (UMAP) in comparison to Principal Component Analysis (PCA). UMAP is an unsupervised nonlinear dimensionality reduction method that is less sensitive to outliers within a data set. It provides a visualization of the separation and overlap among gasoline, kerosene, and diesel samples, allowing analysis of similarities and differences. Through analysis using UMAP it was confirmed that solvent choice substantially affected the chemical profile, with extraction methods for hexane yielded greater cluster separation and reduced intra-class variance, whereas others, such as diethyl ether, showed distortion. This approach demonstrated that UMAP can serve as a diagnostic tool to maximize analyte classification and pattern recognition. By clarifying the similarities and differences of both intra- and inter-sample classes, this approach provided a systematic method for data processing. This work contributes to the development of method workflows for forensic fire debris analysis and supports ongoing efforts to select solvents that minimize exposure of hazards and comply with federal regulations.

P-15

VOLATILE CHARACTERIZATION OF AMERICAN FOULBROOD DISEASE USING COMPREHENSIVE TWO-DIMENSIONAL GAS CHROMATOGRAPHY

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Abstract

American foulbrood (AFB) is a serious disease targeting honey bees that are depended on as livestock and pollinators. Because the *Paenibacillus larvae* bacterial spores that cause infection are very persistent, early identification is necessary to prevent spread to other hives. Diagnosis is generally done through tedious visual inspection that is disruptive to bees, requiring the removal of frames from each hive. In states where AFB is reportable, identification requires euthanasia of the honey bee colony and destruction of the entire hive. Even when no destruction order is present, best practice for infection involves burning all of the frames – an expensive and time consuming process. Such devastation has inspired an earlier detection method for AFB using scent detection dogs. Dogs would allow beekeepers to quickly screen hundreds of hives, identifying infected colonies before the disease has spread throughout the operation, and minimizing the equipment and livestock that would be destroyed.

To understand the odors causing the dogs to alert for AFB, a volatile profile of infected hive material was produced with a nontargeted analysis using comprehensive two-dimensional gas chromatography with time-of-flight mass spectrometry. This profile describes the volatile compounds specific to *P. larvae* as compared to healthy samples. Furthermore, the success of a proposed treatment method using gamma-irradiation was evaluated by inspecting whether the volatile organic compounds associated with *P. larvae* were eradicated in treated samples. This research used solid-phase microextraction arrow, quad-jet dual-stage cryogenic modulation, and analysis with ChromaTOF Sync 2D to produce a detailed characterization of AFB-associated volatiles.

P-16

ADVANCED FUEL AND PETROCHEMICAL ANALYSIS WITH COMPREHENSIVE TWO-DIMENSIONAL GAS CHROMATOGRAPHY (FLOW-MODULATED GCXGC-FID)

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Abstract

Characterizing today's expanding range of energy and chemical feedstocks including conventional jet fuel, Synthetic Aviation Turbine Fuel (SATF), renewable intermediates, and pyrolysis oils, has outpaced the resolving power of conventional one-dimensional gas chromatography (1D-GC). These materials contain overlapping hydrocarbon classes and trace components with wide volatility and polarity ranges, making accurate composition essential for fuel certification, process optimization, and assessment of emerging circular feedstocks.

Comprehensive two-dimensional gas chromatography (GC×GC) provides this capability by pairing two orthogonal columns with a flow modulator that repeatedly refocuses and transfers fractions to the second dimension. The result is a structured chromatogram that clearly resolves paraffins, naphthenes, aromatics, and other key classes across these complex matrices.

PAC powered by AC's Sustain2D Analyzers brings GC×GC performance into routine laboratory workflows. Through robust modulation, optimized column sets, and automated group-type quantification, Sustain2D systems deliver the separation efficiency and data confidence of advanced GC×GC while maintaining everyday operational simplicity. This makes GC×GC a practical, production-ready tool for fuels, petrochemicals, and next-generation sustainable feedstocks.

P-18

MICROPLASTICS AS VECTORS OF ORGANIC CONTAMINANTS ON SOUTHERN CALIFORNIA BEACHES: A TD/PY-GCXGC-TOFMS STUDY

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Abstract

Microplastics (MPs) are pervasive pollutants throughout our local marine environments. They serve as vectors for organic compounds, yet the variability of their adsorbed contaminants along highly urbanized coastlines remains poorly resolved. This study examines the variation in MP contamination and its associated organic compounds across three beach sites in the greater Los Angeles region. Surface sediments will be collected from each site. MPs will be isolated through a density separation to remove inorganic components, followed by oxidative digestion of the remaining organic matter. The recovered MPs will be characterized using an inlet capable of high-temperature thermal desorption/pyrolysis coupled with two-dimensional gas chromatography-time-of-flight mass spectrometry (GCxGC-TOFMS) to identify polymer-specific marker compounds and thereby determine MP composition and relative abundance at each location. In addition, a preliminary thermal desorption (TD) step will be applied before pyrolysis to assess organic compounds adsorbed to MP surfaces. Chromatographic profiles from the single-step TD/PY-GC×GC-TOF-MS analyses will be statistically compared among sites using Chromatof Sync 2D software to evaluate differences in both polymer types and co-occurring contaminants. The results will provide insight into how local sources, hydrodynamics, and land use patterns influence the distribution of MP polymers and their adsorbed contaminant loads along urbanized Southern California coastlines.

P-19

COMPARISON OF SOLID PHASE MICROEXTRACTION COATINGS FOR HEADSPACE EXTRACTION OF VOLATILE PERFLUOROALKYL SUBSTANCES USING ONE- AND TWO-DIMENSIONAL GAS CHROMATOGRAPHY

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Abstract

Perfluoroalkyl substances (PFAS) are used in manufacturing processes due to their oleophobicity, hydrophobicity, and resistance to chemical and thermal degradation. Of growing concern are low-molecular-weight PFAS (<700 Da), a group that encompasses volatile species. Quantification of volatile PFAS is hindered by their volatility and low environmental concentrations. Moreover, accurate detection can be complicated by co-elution in one-dimensional (1D) gas chromatography (GC) and elevated background signals in complex matrices. In this work, two solid phase microextraction (SPME) Arrow coatings, divinylbenzene/carbon-wide range/polydimethylsiloxane (DVB/C-WR/PDMS) and hydrophilic-lipophilic balance/polydimethylsiloxane (HLB/PDMS), were systematically evaluated. The HLB/PDMS coating improved extraction for the polar fluorotelomer alcohols, yielding 706% and 284% increases in peak-area response for 1H,1H,2H,2H-perfluoro-1-hexanol (4:2 FTOH) and 1H,1H,2H,2H-Perfluoro-1-octanol (6:2 FTOH), respectively, relative to DVB/C-WR/PDMS. This enhanced performance motivated full method validation of the HLB/PDMS Arrow in 1D-GC and comprehensive two-dimensional gas chromatography (GC×GC). GC×GC provided substantial analytical improvements. For example, LOQs improved from 0.05 to 0.005 μg L^{-1} (F-hexene) and from 1 to 0.005 μg L^{-1} (10:2 FTOH) from 1D to 2D. Signal-to-noise ratios increased proportionally (e.g., 98 to 344 for F-hexene and 162 to 548 for 10:2 FTOH), demonstrating enhanced resolving power of GC×GC for trace volatile PFAS. Collectively, these results demonstrate that pairing HLB/PDMS SPME with GC×GC markedly improves detection sensitivity, minimizes co-elution, and expands linear dynamic ranges. This combined approach provides a robust platform for the quantitative analysis of volatile PFAS in complex samples, supporting more comprehensive monitoring.

P-20

PAINTING A CLEARER PICTURE: UNTARGETED PERFLUOROALKYL SUBSTANCE DETECTION IN HOUSEHOLD PAINTS USING SOLID PHASE MICROEXTRACTION AND TWO-DIMENSIONAL GAS CHROMATOGRAPHY

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Abstract

Exposure to perfluoroalkyl substances (PFAS) can occur through direct pathways (e.g., application of cosmetics) and indirect pathways (e.g., inhalation of contaminated dust), resulting in multiple points of human exposure. Recent work has shown that paint can be a source of exposure to neutral volatile PFAS such as fluorotelomer alcohols (FTOHs), which can undergo biotransformation into toxic perfluoroalkyl carboxylic acids such as perfluorohexane carboxylic acid. However, no standardized methodology currently exists for the analysis of volatile PFAS in complex matrices. Their determination is further complicated by their trace-level concentrations and propensity to volatilize during conventional sample preparation. Here, we demonstrate the ability of a solid phase microextraction (SPME) Arrow with a hydrophilic-lipophilic balanced (HLB)/polydimethylsiloxane (PDMS) extraction phase to effectively preconcentrate a range of volatile PFAS from paint samples. Paint is a complex matrix with co-eluting compounds; one-dimensional GC lacks the resolving power needed to isolate volatile PFAS from coeluting compounds. Comprehensive two-dimensional gas chromatography (GC×GC) circumvented this bottleneck and enabled confident detection and identification. Analyte identification was assigned using Putative ID[i] and [ii], based on similarity % and calculated retention indices compared to the NIST library. Across Paints 1–5, the number of chemical features assigned Putative ID[i] or [ii] ranged from 230 to 830, with perfluorinated hits representing 0.9–3.0% of identifications. Paint 2 exhibited the highest proportion of perfluorinated features (3.0%), followed by Paints 5 (2.2%) and 1 (1.1%). This workflow can be readily extended to other consumer products, enabling more comprehensive characterization of volatile PFAS sources.

P-21

ALIGNMENT AND FILTERING TOOLS FOR ENHANCED DIFFERENCING OF TWO-DIMENSIONAL CHROMATOGRAMS

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Abstract

Comparative analysis of two-dimensional chromatograms can reveal subtle differences between samples, such as peaks present in one chromatogram but absent in another. A straightforward pixel-to-pixel subtraction of intensities, however, often produces noisy results, fails to account for retention time shifts, and may obscure meaningful low-intensity differences.

We present a chromatogram differencing tool designed to overcome these limitations. The tool enables correction of retention time misalignments through user-defined alignment markers and transform functions. Local variations are accommodated via a fuzzy radius parameter, which provides tolerance in the alignment of compared data points. In addition, normalization options are applied to facilitate comparisons across chromatograms with differing response magnitudes, while advanced filtering suppresses noise and large absolute differences of limited relative significance. This approach enhances the visibility of subtle, low-intensity differences in the final output.

We demonstrate the utility of the tool on both GCxGC and LCxLC-MS datasets, and further illustrate how its alignment functionality can be used to synchronize and fuse MS and FID data acquired from dual-detector systems.

P-22

DEVELOPMENT OF A FULLY SOFTWARE-CONTROLLED, ALIGNMENT-FREE LOOP-DELAY THERMAL MODULATOR FOR GC×GC

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Abstract

We present a significantly simplified and modernized loop-delay thermal modulator for comprehensive two-dimensional gas chromatography (GC×GC). The key innovation is the elimination of stringent loop-alignment requirements, which greatly enhances robustness and ease of use. The design also removes all moving mechanical parts. All modulation parameters—including hot-jet temperature, valve operation, variable modulation period, and cold-jet gas flow—are controlled entirely through dedicated software, independent of the GC instrument. By programming the cold-jet gas-flow rate, the system generates different effective modulation temperatures for compounds of varying volatility.

Two models were developed to address different volatility ranges. The first employs liquid nitrogen (LN_2) to provide maximum cooling power, enabling effective modulation of highly volatile C4+ compounds. The second incorporates an integrated electric chiller, offering cryogen-free operation suitable for the modulation of less-volatile C7+ compounds. Both variants are compatible with most commercial gas chromatographs.

This non-aligned, fully software-controlled loop-delay architecture delivers a robust and flexible solution for high-resolution GC×GC applications, offering a practical modernization of traditional loop-delay modulation.

P-23

THE EFFECT OF USING LOW-BLEED COLUMNS IN THE FIRST DIMENSION FOR COMPREHENSIVE TWO-DIMENSIONAL GAS CHROMATOGRAPHIC ANALYSES

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Abstract

In gas chromatography, the presence of column bleed impacts peak data quality. As the siloxane-based stationary phase fragments, it releases bleed compounds into the mass spectrometer (MS), producing interference and decreasing signal-to-noise ratios (S/N) of analytes. A secondary column with an orthogonal stationary phase is used in comprehensive two-dimensional gas chromatography (GC×GC), providing improved separation between column bleed and analytes, but also increasing the bleed detected. Unfortunately, no prior studies have investigated the effect of low-bleed columns on resulting data from GC×GC-MS analyses.

Therefore, this study explored how replacing the primary column in a GC×GC-MS system impacts the resulting S/N and analyte identification quality. A standard containing approximately 100 different analytes within different classes (i.e Century Mix) and a 52-component Indoor Air Standard were both assessed using a 5% phenyl/95% dimethylsiloxane standard column (first dimension) and a (50%-phenyl)-methylpolysiloxane standard column (second dimension), then re-analyzed with a low-bleed column replacement in the first dimension. Data demonstrated that samples run on the low-bleed column setup exhibited higher S/N compared to samples run on the standard first dimension phase, indicating reliable detection of low concentration analytes. This research clarifies how column bleed impacts analytes that appear to be chromatographically resolved from column artifacts in GC×GC output, and demonstrates the advantage that low-bleed columns can give for future analyses.

P-24

THE CHARACTERIZATION OF POLY(1-BUTENE) VIA PYROLYTIC CONVERSION USING COMPREHENSIVE TWO-DIMENSIONAL GAS CHROMATOGRAHPY HIGH-RESOLUTION TIME-OF-FLIGHT MASS SPECTROMETRY

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Abstract

Pyrolytic conversion of plastic waste into synthetic fuels is a topic of current interest. Pyrolysis combined with comprehensive two-dimensional gas chromatography and high-resolution mass spectrometry (GCxGC-HRMS) is necessary to characterize the highly complex mixtures resulting from polymer pyrolysis. The products of pyrolysis of poly(1-butene) were examined by using a multi-functional pyrolysis system (EGA/PY-3030D, Frontier Lab, Ltd.) and a high-resolution time-of-flight (HRTOF) mass spectrometer (JMS-T2000GC AccuTOF GC-Alpha, JEOL Ltd.) with electron ionization (EI) and photoionization (PI). The GCxGC chromatogram revealed a very complex mixture of products with varying degrees of unsaturation and oxidation. Photoionization produced molecular ions for branched hydrocarbons that did not exhibit them in EI mass spectra. Integration of the EI and PI data was accomplished using a structure analysis tool for unknown compounds that combines GC/EI and GC/soft-ionization high-resolution data with artificial intelligence (AI) structure analysis using four AI technologies (msFineAnalysis AI, JEOL Ltd.). Mass defect analysis of the photoionization data gave an overview that facilitated the identification of compound classes on the GCxGC chromatogram. Fragment ions in the photoionization mass spectra were dominated by fragmentation at branching points.

P-25

DECOMPOSITION ANALYSIS USING DIFFERING DATA PROCESSING METHODS TO IDENTIFY VOLATILE ORGANIC COMPOUNDS

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Abstract

When a body decomposes, it emits volatile organic compounds (VOCs). Decomposition odor has been studied; however, more research is needed to understand VOCs from submerged remains. The decomposition VOC profile can be captured by water in association with the remains. Comprehensive two-dimensional gas chromatography with time-of-flight mass spectrometry (GC×GC-TOFMS) is used to profile these VOCs. GC×GC separates analytes based on their affinity for two column phases separated by a modulator to increase peak capacity for complex samples. Nine mason jars filled with pork belly and tap water were placed in three different temperature conditions (32°C, 22°C, ~5°) in triplicate, including controls that contained only water. The time trial lasted 12 days with 5 mL of water sampled from the jars each day until day 3, then every third day until day 12. Samples were analyzed using headspace solid phase microextraction (SPME) arrow and GC×GC-TOFMS with dual-stage cryogenic modulation. Data was processed using peak table-based software, batch alignment software, and tile-based software. The software approaches were compared to determine which program is best suited for forensic investigation based on accuracy of analyte identification, representation of decomposition over time, and effective class characterization. The batch alignment software showed promise for forensic research for longitudinal data to show trends in VOCs. The tile-based class differentiation software was best to compare samples from submerged remains to water containing none. This type of comparison can provide forensic scientists with better understanding of decomposition VOC uses for research and the field.

P-26

EFFECT OF COLUMN LENGTH ON THE RESOLVING POWER OF SEPARATIONS OF NON-IONIC SURFACTANTS BY TWO-DIMENSIONAL LIQUID CHROMATOGRAPHY

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Abstract

Non-ionic surfactants manufactured for consumer applications are highly complex mixtures containing thousands of different chemical species. Starting in the 1990's the utility of two-dimensional liquid chromatography (2D-LC) for unravelling these complex mixtures was demonstrated, and has been used to great effect in the decades since. In this presentation we will discuss the effect of column length on the resolving power of these separations. The majority of such separations described in the peer-reviewed literature describe the use of conventional column lengths (e.g., 50 to 150 mm) in both separation dimensions. Since the first dimension separation is typically operated at sub-optimal (in a van Deemter sense) flow rates, there is ample opportunity to explore the possibility that longer columns might yield higher resolving powers. We have systematically studied the effect of column length on peak capacity of separations suitable for use as the first dimension in 2D-LC separations of surfactants such as diblock copolymers of ethylene- and propylene oxide containing a fatty alcohol end group. We find that the peak capacity roughly increases in proportion to the square root of column length, as expected from chromatographic theory. However, this benefit comes at the considerable cost of purchasing additional columns to increase the length, since columns exceeding 250 mm are not widely available for liquid chromatography. Nevertheless, we hope these results will inspire further research in this area, where commercial offerings of much longer columns would be very beneficial.