Developments Gas Chromatography to Gas Chromatography × Gas Chromatography

Alireza Ghassempour
Prof. of Anal. Chem.
Medicinal Plants and Drugs Research Institute
Shahid Behshti University

Dec. 4. 2013
The gas chromatographic system used in Prior’s work, in 1945-1947. A = adsorbent for purification of the carrier gas (hydrogen); B = sample inlet system; C = buret containing mercury with niveau glass for sample introduction; D = Dewar flask; E = separation column (containing silica gel on activated carbon); and F = thermal conductivity detector.
Two Dimensional Gas Chromatography
<table>
<thead>
<tr>
<th>Year Range</th>
<th>Event</th>
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<tbody>
<tr>
<td>1955</td>
<td>First commercial instrument (thermal conductivity detection)</td>
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</table>
| 1955-1960  | Rapid growth in technology  
*Invention of FID, coupling to MS and Temperature Programming* |
| 1960-1970  | Period of technical advances  
*Introduced first open-tubular columns, Transistors replace vacuum tubes*  
*Improvements in detector technology (TID, FPD and ECD)* |
| 1970-1980  | Period of consolidation and refinement  
*Introduced microprocessor-based, Computing integrators, Fused-silica open-tubular columns* |
| 1980-1990  | Period of technical advances  
*Developed Gum and immobilized phases, Wide-bore open-tubular columns, Fundamenta*  
*Fundamental basis for injection (On-column, PTV, Large-volume, Autosamplers)* |
| 1990-2000  | Period of consolidation and refinement  
*PC control, Electronic pneumatic control, Developed new Detectors SPME, chip, Portable GC* |
| 2000-present | Modified GC characteristics and parameters  
*Fast GC, Stir bar and new absorbants for TD, 2D-GC* |
This chart shows the total number of documents for this query by Year.

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SCOPUS, Dec. 3, 2013
GC Determination of Oxygenates in Gasoline

Comparison between Multidimensional GC and Oxygen Selective Flame Ionization Detection (O-FID)

Thermo Fisher Scientific Inc., Milan, Italy

Introduction

The stringent requirement for reducing automobile emissions and improving air quality calls for replacing the usual anti-knock compounds added to gasoline to enhance the octane values with oxygenated compounds that feature a lower toxic impact on the environment. Type and concentration of ethers, alcohols, and other various oxygenates are specified and regulated to ensure acceptable commercial gasoline quality [1]. In this scenario, the ability to monitor the content of this category of compounds in gasoline in an easy and reliable manner is of great importance.

This application compares two different ASTM compliant methodologies used to determine the content of oxygenates in gasoline, both implemented on the Thermo Scientific TRACE GC Ultra™ platform (Figure 1): a multidimensional method according to ASTM 4815, and an oxygen selective detector-based method (O-FID) according to ASTM 5599.

Figure 1: TRACE GC Ultra with Valve Oven

The sample is injected through a Split/Splitless (S/SL) injector. After the elution of Methyl Cyclopentane, but provides the separation of the oxygenates according to their boiling points. The remaining heavier components are then eluted prior to the start of a new analytical cycle by extending the overall run time to approximately 18 minutes. ASTM D4815 describes and allows the use of two alternative detection systems: Thermal Conductivity (TCD) or Flame Ionization (FID). This application involves the use of a TCD detector.

Figure 2: Schematics used to apply method ASTM 4815

ASTM 4815: Analytical Process

The sample is injected through a Split/Splitless (S/SL) injector. After the elution of Methyl Cyclopentane, but
Review

Qualitative and quantitative approaches in comprehensive two-dimensional gas chromatography

Jacolin A. Murray

Analytical Chemistry Division, National Institute of Standards and Technology, 100 Bureau Dr., Mail Stop 8392, Gaithersburg, MD 20899-8392, USA

ARTICLE INFO

Article history:
Available online 9 May 2012

Keywords:

ABSTRACT

Comprehensive two-dimensional gas chromatography (GC × GC) offers advantages over one-dimensional gas chromatography (GC) including, high peak capacity, signal enhancement, and structured chromatograms. These advantages have been exploited to solve several analytical problems that are difficult to achieve in GC. In this review, qualitative and quantitative approaches of GC × GC are explored, including...
**Metabolome** = the total metabolite pool

- All low molecular weight (MW < 1000 Da) organic molecules in a sample such as a leaf, fruit, or tuber.
Peak apex plots corresponding to Separation of apple constituents. On GC × GC system employing the Rxi-5SilMS column in the first dimension volatility-based separation and A – Supelcowax and B – DB-17 columns in second dimension. S/N and similarity thresholds were set to 50 and 800, respectively.
Chromatograms obtained in the pre-column of the MDGC system from either direct introduction of C. carvi seeds (a) or injection of C. carvi essential oil (b) and stereo differentiations achieved in the main column (c and d) of limonene and carvone by transferring, from the first to the second dimension, the indicated cuts resulting from the pre-column separations.
22 years ago, Comprehensive Two-Dimensional Gas Chromatography (GC-GC) introduced by John Phillips and Zaiyou Liu in 1991.
Apparatus and method of multi-dimensional chemical separation

Patent number: 5196039

Abstract: A practical device and method of performing comprehensive multi-dimensional chemical separation using a first dimension of a two-dimensional chromatograph to generate a chromatogram in a time comparable to or even faster than common practice while the second dimension generates multiple chromatograms each in a time comparable to the fastest prior art chromatography. The transfer of sample portions from the first dimension to the second dimension is by any one of several sample stream modulation techniques. These techniques accumulate portions of sample between the first and the second dimensions transferring them as very sharp concentration pulses analogous to fast injections, without loss of sample and with a substantial improvement in sensitivity.

Type: Grant

Filed: March 6, 1992

Issued: March 23, 1993

Assignee: Southern Illinois University at Carbondale

Inventors: John B. Phillips, Zaiyou Liu
John Phillips: Patents

**Apparatus and method of multi-dimensional chemical separation**
Southern Illinois University at Carbondale
John B. Phillips, Zaiyou Liu
A practical device and method of performing comprehensive multi-dimensional chemical separation using a first dimension of a two-dimensional chromatograph to generate a chromatogram in a time comparable to or even faster than common practice while the second dimension generate...

View Patent

**Chromatographic technique and apparatus**
The Board of Trustees of Southern Illinois University
John B. Phillips, Zaiyou Liu
To provide a compact, sensitive comprehensive, two-dimensional gas chromatograph, a chromatographic column has first and second sections in series with each other to permit the flow of sample and a carrier through the first section and the second section. The retention time of...

View Patent

**Apparatus and method for chemical modulation**
Board of Trustees of Southern Illinois University on Behalf of Southern Illinois University at Carbondale
Edward B. Ledford, Jr., John B. Phillips
An apparatus and method are described for forming a chemical modulation of a substance present in a fluid stream, which utilize a movable device, such as a movable heater, to induce changes in the retention of a chemical substance flowing through the modulator tube. The modu...

View Patent
Multidimensional Gas Chromatography: Past, Present & Future

Philip Marriott
School of Chemistry, Monash University

Discovery Outstanding Researcher Award: Australian Research Council
Acknowledgements

GC×GC Co-Chairs:
Jean-Marie Dimandja (USA)
Philip Marriott (Australia)

GC×GC Scientific Committee:
Jean-Marie Dimandja (USA)
Hans-Gerd Janssen (The Netherlands)
Philip Marriott (Australia)
Luigi Mondello (Italy)
John Seeley (USA)
Robert E. Synovec (USA)
Presenter Biographies

Prof. Uwe J. Meierhenrich
University of Nice-Sophia Antipolis, France

Uwe J. Meierhenrich studied chemistry at the Philipps University of Marburg. After completing his Ph.D. at the University of Bremen, he identified amino acids in artificial comets at the Max Planck Institute for Solar System Research in Katlenburg-Lindau and at C.B.M. in Orleans in preparation for the cometary Rosetta mission. Since 2005, he is a full Professor at the University of Nice-Sophia Antipolis. Meierhenrich published his most recent book Amino Acids and the Asymmetry of Life with Springer-Verlag in 2008. His gas chromatographic equipment for the multidimensional and enantioselective analyses of amino acids is also appreciated and applied for the analysis of aroma and perfume compounds. Having awarded the Hans Poppewski Prize from the GCxGCh in 2011 for his...
37th International Symposium on Capillary Chromatography
Frantisek Svec, Lawrence Berkeley National Laboratory
Robert E. Synovec, University of Washington

10th GC×GC Symposium
Jean-Marie Dimandja, Spelman College
Philip Marriott, Monash University

May 12-16, 2013
Renaissance Palm Springs
Palm Springs, CA USA
Simultaneous Analysis for Complex PAH Mixtures Using Novel Column Combinations in GC×GC/TOF-MS
Eunha Hoh¹; Carlos Manzano²; Staci Massey Simonich²,¹San Diego State University, San Diego, CA USA; ²Oregon State University, Corvallis, OR USA

Identifying Biomarkers of P. aeruginosa Antibiotic Susceptibility Using GC×GC-TOFMS and Fisher Ratios
Heather Bean¹; Jean-Marie D. Dimandja²; Jane E. Hill¹;¹University of Vermont, Burlington, VA USA; ²Spelman University, Atlanta, GA USA

Development of Nitrogen Chemiluminescence as a Powerful Detector for GC×GC
Jacqueline Hamilton¹; Mustafa Z. Özel¹; Noelia Ramirez¹; Alastair C. Lewis²; Emanuela Finessi¹;¹University of York, York, United Kingdom; ²NCAS, University of York, York, United Kingdom

Separation and Identification of 'Supercomplex' Mixtures of Toxic Organic acids by GC×GC-High Resolution-MS with Ionic Liquids: a 'Hump' No More!
Steven Rowland, Plymouth University, Plymouth, United Kingdom

GC×2GC and 2GC×GC using Contra-Directional Thermal Modulation
Benjamin Savareear¹; Laura Tedone²; Robert Shellie¹;¹ACROSS, University of Tasmania, Hobart Australia; ²University of Messina, Messina Italy

Taking a Good Dose of High Separation Medication for Gas Chromatography Analysis of Fatty Acid Methyl Esters
Philip Marriott; Annie Zeng; Asia Nosheen; Yada Nolvachai; Blagoj Mitrevski; Sung-Tong Chin, Monash University, Clayton, Australia

Optimization of Column Formats and Flow Conditions in GC×GC
Hans-Gerd Janssen¹; Daniela Peroni²;¹Unilever, Vlaardingen, The Netherlands; ²University of Amsterdam, Amsterdam, The Netherlands
His main research activities are in Analytical Chemistry, specifically gas chromatography (multidimensional GC and comprehensive 2D GC) with mass spectrometry.
**Current Projects**

1. LC, GC and MS of surfactants
2. Illicit drugs analysis; Personal care / pharmaceutical / forensic drugs analysis
3. Food and Beverage analysis; Fatty acids profiling
4. Essential oils analysis; Olfactometry; Wine; Coffee and related samples
5. Theories and simulations of GCxGC data
6. Derivatisation and GCxGC of flavonoids, plant phenols, plant hormones and related plant-derived chemicals
7. Analysis of traditional Chinese medicines and herbals
8. Studies in Multidimensional GC and Preparative GC with Spectroscopy
9. Chirality in chemical separations

**New Projects**

*We have new funding support for research in the areas of:*

1. Analysis of Petroleum products
2. MDGC with Spectroscopy for Absolute Chemical Characterization
3. Illicit and Sports Drugs Analysis
4. Study of Polymer surface coatings in capillary GC
The complexity of GC-GC related data and high-throughput analysis for real mixtures make chemometrics widely applicable to this area!

• First, chemometrics methods to deconvolute overlapping GC-GC peak clusters in 1D and 2D separations are introduced by using model or fitting techniques. Based on the 2D feature of GC-GC separation, conventional deconvolution methods for 2D or 3D data processing have been applied for GC-GC processing.

• Second, MCR methods based on single or multiple runs are separately summarized, to extract chromatographic data and spectral profiles of pure components from GC-GC-TOF-MS data to support identification and quantification.

• Four important chemometrics methods for 2-way and 3-way data resolution are introduced in theory, with worked examples of processed GC-GC-related data, including heuristic evolving latent projection (HELP), parallel factor analysis (PARAFAC), MCR-alternating least squares (MCR-ALS), and alternative moving window factor analysis (AMWFA).
real problems

① experimental optimization for sample extraction and GC×GC analysis

② signal processing for data preprocessing and information extraction

chemometrics

③ MCR for identification and quantification
④ PR for clustering and discriminant analysis
⑤ quantitative modeling for QSAR/QSPR study
⑥ new insights of GC×GC study (orthogonality, image analysis, etc.)

results interpretation
Jeremy Samuel Arey

Environmental Chemistry Modeling Laboratory, EPFL

He employs a variety of computational and experimental methods, including Molecular Dynamics simulations, Comprehensive Two-Dimensional Gas Chromatography - Time-of-Flight Mass Spectrometry (GCxGC-TOFMS) analysis, and other cutting edge tools.

- His philosophy is to draw upon fundamental chemical and physical concepts to construct quantitative descriptions of chemical behavior in aquatic environments.
- They test and validate our approximations and computational methods using independent experimental data, field measurements, and high quality theoretical calculations.
Companies
<table>
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<tr>
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Separate complex mixtures without complex hardware

A number of different modulator designs exist, mostly relying on thermal cycling to focus the bands from the first column and release them into the second column. There are some disadvantages to this approach:

- Large usage of expensive cryogenic gases leading to a high cost of analysis
- Complexity of the hardware
- Longer analysis times

Agilent’s proprietary Capillary Flow Technology and further-generation Electronic Pneumatics Control (EPC) enable the use of a differential flow modulator to conduct comprehensive GCxGC without the use of cryogenic gases or complex hardware.

How it works: the modulator is key

The capillary Flow Technology modulator uses a deactivated, stainless steel structure with all flow splitters and the collector channel incorporated internally in the device. With its low thermal mass it can track even temperature very closely, while its GC oven location allows precise temperature control without lag during programmed runs. All external connections are made using Agilent’s Scott Union technology for leak-free operation and extremely small, well-sealed volumes. An in-line three-way solenoid valve, installed on the side of the gas chromatograph, connects to a pneumatics control module (PCM) to accurately and precisely control flow through the modulator.

![Diagram of the modulator](image)

**Figure 1.** Load in cold state (above). At the beginning of the scan, the collection channel is filled with hydrogen from a previous injection cycle. The previous column (bottom) contains a modulator to capillary and focus the bands into the collection channel. The column then feeds to the second column and the collection channel. Hydrogen from the previous column (top) fills the modulator with bottom gas and enters the second column.
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- Simplifies column selection for method developers and heavy GCxGC users alike.
- Press-Tight® connectors also included.

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LECO’s GCxGC
Comprehensive Two-Dimensional Gas Chromatography

Take your chromatography to the next level

LECO comprehensive two-dimensional gas chromatography (GCxGC) with FID and/or ECD detectors is the ideal solution for your analytical methods which require superior resolving power over that which you have been able to achieve with traditional GC. Whether you need to speciate sulfur-containing compounds in petroleum, separate halogenated pesticides, or quantify chiral compounds in flavors or fragrances, LECO's GCxGC options deliver unparalleled separating power and up to an order-of-magnitude increase in signal to noise.
<table>
<thead>
<tr>
<th>Company</th>
<th>Part/Instrument</th>
<th>Site</th>
</tr>
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GC x GC Schematic

- Injector
- Loop Modulator
- Primary Column
- Secondary Column
- Detector
- Secondary Oven
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</table>
- FID Detector
- Dual On-Column Injectors with Pressure Switching
- Dual Capillary Columns (1 polar, 1 non-polar)
- Built-in “whisper quiet” Air Compressor
- 1 channel PeakSimple Data System
- on the 8610D Dual Oven chassis

The 2D GC System is similar to the TCS GC, but it also permits true comprehensive 2D separations using pressure switching and modulation. The Two Dimensional GC system allows users to modulate the effluent from the first column, which is non-polar, onto a second column, which is polar, in a series of short chromatographic slices. The polar column may be operated isothermally, or it may be temperature programmed. Peaks which elute from the non-polar column may sometimes be separated more effectively in two dimensions than in one dimension,
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Merge of GC$x$GC and GC-MS
<table>
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<tr>
<th>Separation Speed</th>
<th>Column i.d. (mm)</th>
<th>Column Length (m)</th>
<th>Theoretical Plates</th>
<th>Retention Time (s)</th>
<th>Peak Width 2.354σ (s)</th>
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<tbody>
<tr>
<td>Standard</td>
<td>0.32</td>
<td>25</td>
<td>75,000</td>
<td>100</td>
<td>0.7</td>
</tr>
<tr>
<td>Fast</td>
<td>0.05</td>
<td>10</td>
<td>260,000</td>
<td>60</td>
<td>0.2</td>
</tr>
<tr>
<td>Very fast</td>
<td>0.05</td>
<td>1</td>
<td>24,000</td>
<td>2</td>
<td>0.03</td>
</tr>
<tr>
<td>Ultrafast</td>
<td>0.05</td>
<td>0.3</td>
<td>6,500</td>
<td>0.3</td>
<td>0.01</td>
</tr>
</tbody>
</table>

![Mass spectrum acquisition rate diagram](image_url)
The physical laws governing mass selection processes limit the ability of quadrupole mass analyzers to achieve very fast data acquisition rates. Contemporary quadrupole MS instruments are capable of mass scanning rates of around 10,000 amu/s. Thus, for a full-scan mass range of 50 to 350 amu, the maximum spectral acquisition rate would be expected to be less than 30 spectra/s. Very fast GC is best mated with nonscanning TOF-MS, which is capable of very fast full-spectrum data acquisition rates (50 spectra/s will suffice for fast GC).

Although, there are some examples of ultrafast second-dimension GC × GC separations in the literature, these are outside the norm, and 50 to 100 spectra/s is generally applicable for GC × GC–MS analysis.
Scan rates
(A) Experimental setup of the gas chromatography SPI–oa–TOF MS instrument. SPI is performed in a separate ionization chamber with 126-nm photons from an Ar-filled EBEL VUV lamp. (B) Comprehensive two-dimensional GC × MS representation obtained from a gas chromatographic analysis of a diesel sample with an EBEL VUV lamp for SPI. (C) If GC × GC is combined with soft SPI–TOF MS, a three-dimensional comprehensive separation can be realised with the separation axes first and second retention time and molecular mass (GC × GC × SPI–MS).
Acknowledgment:

1. Dr. Zahra Talebpour
2. Mr. Mostafa Alilou
3. Mrs. Rihaneh Safavi
4. Mrs. Farideh Haghighi