

Comprehensive Two-Dimensional High-Speed Gas Chromatography with Chemometric Analysis

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High-speed comprehensive two-dimensional gas chromatography (GC $\times$ GC) is performed, in which a polar second column performs separations every half second on portions of the effluent from a nonpolar first column. Chemometric techniques that are traditionally used on chromatographic separations with multichannel detection are applied to two-dimensional chromatographic data, for the purpose of quantifying incompletely resolved peaks. Generalized rank annihilation method (GRAM) is evaluated in the quantification of varying amounts of selected overlapped analytes in a GC $\times$ GC analysis of modified white gasoline. GRAM requires a sample and standard data set for quantification, and the high retention time precision arising from use of shortened GC columns aids in the analysis. Results from GRAM analysis of GC $\times$ GC data are compared with a reference GC method. The test analytes ethylbenzene and *m*-xylene, existing in various proportions in white gasoline samples, were successfully deconvoluted despite having resolutions of 0.46 and 0.20 on the first and second dimensions of separation, respectively. Like other second-order techniques, GRAM was able to reliably quantify *m*-xylene despite the presence in the analytical sample of an overlapping compound not present in the calibration standard. Because GRAM can be successfully applied to GC $\times$ GC data, full resolution of all the analytes of interest is not necessary. As a result, GC $\times$ GC run times can be dramatically shortened, which has significant implications for analyses in which short cycle times are critical, such as in process analysis.

Gas chromatographic (GC) analysis of multicomponent samples can be a time-consuming process. To reduce analysis times, many separations are done using multicolumn GC. In traditional multicolumn GC (GC-GC), the first column performs a partial separation of a sample, and a fraction, or heartcut, of the eluent is injected into the second column for final separation. The first column effectively performs sample preparation, reducing the number of compounds injected into the second column. The second column must have a different chemical selectivity than the first column in order to optimize the GC-GC resolving power. The difference in the chemical selectivity between the two columns allows compounds that are poorly separated in the first dimension to be more fully resolved in the second dimension. Although this technique is proven to have enormous practical

value,¹ it can be a time-consuming process. Heartcuts from the first column range from seconds to minutes in length, and the separation that the first column has performed on the compounds within that heartcut is generally undone during the injection onto the second column. The lengthy runtimes of the second column limit the number of heartcuts that can be taken from the first column.

Traditional GC-GC is not a true "hyphenated" technique. Hyphenated chromatographic techniques, such as liquid chromatography with diode array absorbance detection, offer the advantage of being able to identify eluting compounds through inspection of the spectral information. For hyphenated techniques, a vector of data is obtained at short time intervals during a separation, producing a two-dimensional data array for each run. The ideal GC-GC instrument is one that can function as a hyphenated instrument, for it would preserve all the chromatographic information from both dimensions of separation. This ideal system would perform a second dimension separation of an infinite number of infinitely thin heartcuts from the first column. Under these circumstances, the separating power of the ideal system is proportional to the product of the separating powers of the individual columns.^{1,2} There is a practical limit to how frequently a sample can be cut to the second column, so the actual separating power is less than that theoretically calculated. This mode of operation is termed comprehensive GC-GC, or GC $\times$ GC,^{3,4} with the multiplicative term emphasizing the enhanced separation power of this technique. Because GC $\times$ GC can provide much larger peak capacities, difficult separation problems are more easily handled with GC $\times$ GC. In addition, if a separation problem requires a given peak capacity, GC $\times$ GC is capable of generating it much more rapidly than an existing multiple-column GC using a few heartcuts. This article investigates how chemometric techniques can be applied to GC $\times$ GC to get the most information from the data and reduce run times, thus broadening the scope of GC $\times$ GC.

In fast GC, peak width, and thus separation efficiency, is frequently limited by the performance of the injector. To reduce separation times, the injected sample pulse width must be as short in time as possible.⁵ GC $\times$ GC is most useful when the second

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Comprehensive Two-dimensional Gas Chromatography Time-of-flight Mass Spectrometry with Chemometric Analysis Amanda Elizabeth Moses Sinha, 2004 *Chemometric Analysis of Comprehensive Two-dimensional Liquid Chromatographic-diode Array Detection Data* Hope Patricia Bailey, 2012 This research project sought to explore compare and develop chemometric methods with the goal of resolving chromatographically overlapped peaks through the use of spectral information gained from the four way data sets associated with comprehensive two dimensional liquid chromatography with diode array detection LC x LC DAD A chemometric method combining iterative key set factor analysis IKSFA and multivariate curve resolution alternating least squares MCR ALS was developed In the section of urine data analyzed over 50 peaks were found with 18 visually observable and 32 additional compounds found only after application of the chemometric method Upon successful chemometric resolution of chromatographically overlapped peaks accurate and precise quantification was then necessary Of the compared methods for quantification the manual baseline method was determined to offer the best precisions Of the 50 found peaks from the urine analysis 34 were successfully quantified using the manual baseline method with percent relative standard deviations ranging from 0.09 to 16 The accuracy of quantification was then investigated by the analysis of wastewater treatment plant effluent WWTPE samples The chemometrically determined concentration of the unknown phenytoin sample was found to not exhibit a significant difference from the result obtained by the LC MS MS reference method and the precision of the IKSFA ALS method was better than that of the precision of the LC MS MS analysis Chromatographic factors data complexity large dynamic range retention time shifting chromatographic and spectral peak overlap and background removal were all found to affect the quantification results The last part of this work focused on rapid screening methods that were capable of locating peaks between samples that exhibited significant differences in concentration The aim here was to reduce the amount of data required to be resolved and quantified to only those peaks that were of interest This would then reduce the time required to analyze large complex samples by eliminating the need to first quantify all peaks in a given sample for many different samples Both the similarity index SI method and the Fisher ratio FR method were found to fulfill this requirement in a rapid means of screening fifteen wine samples *Comprehensive Chemometrics* Steven Brown, Roma Tauler, Beata Walczak, 2020-05-26 *Comprehensive Chemometrics Second Edition* Four Volume Set features expanded and updated coverage along with new content that covers advances in the field since the previous edition published in 2009 Subject of note include updates in the fields of multidimensional and megavariate data analysis omics data analysis big chemical and biochemical data analysis data fusion and sparse methods The book follows a similar structure to the previous edition using the same section titles to frame articles Many chapters from the previous edition are updated but there are also many new chapters on the latest developments Presents integrated reviews of each chemical and biological method examining their merits and limitations through practical examples and extensive visuals

Bridges a gap in knowledge covering developments in the field since the first edition published in 2009 Meticulously organized with articles split into 4 sections and 12 sub sections on key topics to allow students researchers and professionals to find relevant information quickly and easily Written by academics and practitioners from various fields and regions to ensure that the knowledge within is easily understood and applicable to a large audience Presents integrated reviews of each chemical and biological method examining their merits and limitations through practical examples and extensive visuals Bridges a gap in knowledge covering developments in the field since the first edition published in 2009 Meticulously organized with articles split into 4 sections and 12 sub sections on key topics to allow students researchers and professionals to find relevant information quickly and easily Written by academics and practitioners from various fields and regions to ensure that the knowledge within is easily understood and applicable to a large audience

Advances in Chromatography, Volume 50 Eli Grushka, Nelu Grinberg, 2012-01-27 For more than four decades scientists and researchers have relied on the Advances in Chromatography series for the most up to date information on a wide range of developments in chromatographic methods and applications For Volume 50 the series editors have invited established well known chemists from across the globe to offer cutting edge review

Comprehensive Chemometrics, 2009-03-09 Designed to serve as the first point of reference on the subject Comprehensive Chemometrics presents an integrated summary of the present state of chemical and biochemical data analysis and manipulation The work covers all major areas ranging from statistics to data acquisition analysis and applications This major reference work provides broad ranging validated summaries of the major topics in chemometrics with chapter introductions and advanced reviews for each area The level of material is appropriate for graduate students as well as active researchers seeking a ready reference on obtaining and analyzing scientific data Features the contributions of leading experts from 21 countries under the guidance of the Editors in Chief and a team of specialist Section Editors L Buydens D Coomans P Van Espen A De Juan J H Kalivas B K Lavine R Leardi R Phan Tan Luu L A Sarabia and J Trygg Examines the merits and limitations of each technique through practical examples and extensive visuals 368 tables and more than 1 300 illustrations 750 in full color Integrates coverage of chemical and biological methods allowing readers to consider and test a range of techniques Consists of 2 200 pages and more than 90 review articles making it the most comprehensive work of its kind Offers print and online purchase options the latter of which delivers flexibility accessibility and usability through the search tools and other productivity enhancing features of ScienceDirect

Comprehensive Two Dimensional Gas Chromatography Lourdes Ramos, 2009-07-22 The book reviews the basic concepts and highlights the most relevant advances and developments that have taken place in the field of comprehensive two dimensional gas chromatography GC x GC since its introduction in 1991 The several instrumental and technical approaches assayed and developed during these seventeen years and that have contributed to the development of this powerful separation technique and to its increasing application in many areas is explained and comprehensively

illustrated through a number of chapters devoted these specific topics More specialized aspects of the technique including theoretical aspects modelization of the chromatographic process software developments and alternative couplings is also covered Finally special attention is paid to data treatment for both qualitative and quantitative analysis This book will be a practical resource that will explain from basic to specialized concepts of GC x GC and will show the current state of the art and discuss future trends of this technique Outlines basic concepts and principles of GCxGC technique for non specialists to apply the technique to their research Provides detailed descriptions of recent technical advances and serves as an instructional guide in latest applications in GCxGC Sets the scene for possible future development and alternative new applications of technique Fundamentals and Analytical Applications of Multiway Calibration ,2015-08-10 Fundamentals and Analytical Applications of Multi Way Calibration presents researchers with a set of effective tools they can use to obtain the maximum information from instrumental data It includes the most advanced techniques methods and algorithms related to multi way calibration and the ways they can be applied to solve actual analytical problems This book provides a comprehensive coverage of the main aspects of multi way analysis including fundamentals and selected applications of chemometrics that can resolve complex analytical chemistry problems through the use of multi way calibration Includes the most advanced techniques methods and algorithms related to multi way calibration and the ways they can be applied to solve actual analytical problems Presents researchers with a set of effective tools they can use to obtain the maximum information from instrumental data Provides comprehensive coverage of the main aspects of multi way analysis including fundamentals and selected applications of chemometrics **Advances in Chromatography** Phyllis R. Brown,Eli Grushka,2003-01-14 Illustrating developments in separation science and chromatographic analysis this volume investigates trends in chemometrics proteomics column technology and element selective detection for pharmaceutical medical industrial and environmental applications Advances in Chromatography Nelu Grinberg,Peter W. Carr,2022-12-30 For six decades scientists and researchers have relied on the Advances in Chromatography series for the most up to date information on a wide range of developments in chromatographic methods and applications The clear presentation of topics and vivid illustrations for which this series has become known make the material accessible and engaging to analytical biochemical organic polymer and pharmaceutical chemists at all levels of technical skill Describes the thermodynamics and kinetics underlying hydrophobic interaction chromatography of proteins Outlines use of a kinetic model in the predictive modeling of evaporation processes that eliminates the need to know the composition and identity of the chemical constituents in the sample Explores building and employing QSRR models in cyclodextrin modified high performance liquid chromatography HPLC Reviews chemometric methods commonly paired with comprehensive 2D separations and key instrumental and preprocessing considerations Advanced Chemometric Techniques for the Analysis of Complex Samples Using One- and Two-dimensional Gas Chromatography Coupled with Time-of-flight Mass Spectrometry Brooke C. Reaser,2017 Gas

chromatography is a powerful separation technique that alone and when coupled with mass spectrometric detection can provide detailed information regarding the chemical composition of complex mixtures. Advanced chemometric algorithms are often applied to the data generated from these gas chromatographic separations in order to glean additional meaningful information from large and complex data sets. This dissertation presents several research investigations conducted on the development, optimization, application and study of several chemometric algorithms applied to one and two dimensional gas chromatography coupled with time of flight mass spectrometry (TOFMS). The two dimensional mass cluster method and principal component analysis (PCA) were applied to a non-targeted investigation of the stable isotope incorporation of metabolites present in the metabolome of the methylotrophic bacteria *Methylobacterium extorquens* AM1 using gas chromatography time of flight mass spectrometry (GC TOFMS). The area under the curve (AUC) of receiver operating characteristic (ROC) curves were used as quantitative metrics for the optimization of the tile based Fisher ratio method using diesel fuel spiked with native and non native analytes using comprehensive two dimensional gas chromatography with time of flight mass spectrometry (GC GC TOFMS). This optimized algorithm was then applied to a process analytical chemistry (PAC) investigation into the source of catalyst yield reduction in an industrial polymerization plant. Finally a GC TOFMS simulation based study determined the chemometric limit of resolution for deconvoluting analytes using multivariate curve resolution alternating least squares (MCR ALS) and compared the results to expected theory surrounding the probability of peak overlap.

Advanced Chemometrics and Fundamental Considerations for Non-targeted Analysis with Comprehensive Multidimensional Gas Chromatography Coupled with Time-of-flight Mass Spectrometry Sarah Elizabeth Prebihalo, 2020. Comprehensive two dimensional gas chromatography (GCxGC) coupled with time of flight mass spectrometry (TOFMS) is a powerful analytical technique capable of separating complex mixtures providing valuable information about the chemical composition of samples. However the inherent data density associated with three dimensional data provides a unique challenge to analytical chemists. As a result significant effort has been invested in utilizing advanced chemometrics to glean meaningful information about samples from large and complex data sets. Herein this dissertation introduces several investigations conducted on optimizing separation conditions to be amenable to chemometric deconvolution algorithms as well as the development study and application of advanced chemometric techniques applied to GCxGC TOFMS data. To begin the metric trilinear deviation ratio (TDR) is utilized to study the impact of experimental parameters such as column selection and modulation period (PM) on the quantitative accuracy of parallel factor analysis (PARAFAC) deconvolution. TDR scales with increasing change in second dimension retention time ($\Delta 2t_R$) associated with pseudo isothermal conditions on the second dimension (2D) and quantitative accuracy decreases as TDR increases. Two column sets were utilized with varying film thickness on the first column (1D) and each column set was studied using two PM for a total of 4 experiments. It was reported that using 1D columns with larger film thicknesses allows the analyst to employ a shorter PM in turn lowering the $\Delta 2t_R$.

leading to higher quantitative accuracy Many GCxGC TOFMS studies relate to identifying class distinguishing analytes and can be tedious when performed manually Fortunately the use of discovery based chemometric tools such as principal component analysis PCA and Fisher ratio F ratio analysis has increased in popularity as less time intensive and automated techniques for untargeted analyses To begin this dissertation will investigate mass channel purity obtained via the tile based F ratio algorithm using diesel fuel spiked with non native analytes using GCxGC TOFMS The F ratio algorithm considered a supervised discovery technique because class membership is known a priori was first used to discover the spiked non native analytes Then using a novel signal ratio S ratio algorithm the mass channel selectivity information output by the F ratio method was studied using three statistical metrics null distribution analysis p value and lack of fit LOF The result of this investigation revealed that a mass channel has a high likelihood of being pure when its p value and LOF are sufficiently low Finally F ratio analysis was applied to a dataset including patients with an anterior cruciate ligament ACL injury to discover potential biomarkers of post traumatic osteoarthritis PTOA post injury Standard F ratios are calculated by the between class variance divided by the sum of the within class variance scaling up as the between class variance increases and the within class variance remains sufficiently small However many biological studies involve significant biological variance 30% that may not be associated with disease state or injury severity etc Herein the standard tile based F ratio algorithm was modified to use only the within class variance associated with control samples It was expected that the control class contained less within class variance relative to the patient class due to the expectation that some patient samples would be associated with increased severity of injury or the presence of coexisting conditions Hit lists metabolites discovered via F ratio from standard F ratio and control normalized F ratio were studied and directly compared to establish a comprehensive metabolome of potential biomarkers for PTOA development post ACL injury Reported in this dissertation is a discussion on the complementary nature of standard and control normalized F ratio followed by demonstration of class distinguishing metabolites via PCA *Journal of Chromatography* ,2003 Dissertation Abstracts International ,2008 **American Laboratory** ,2006 **Two-Dimensional Liquid Chromatography** Oliver Jones,2020-07-13 This book addresses the growing interest in the field of two dimensional liquid chromatography 2DLC a powerful approach to increasing resolution available peak capacity and selectivity in analytical chromatography 2DLC is suitable for many applications including in the pharmaceutical and polymer industries and the omic sciences metabolomics lipidomics and proteomics Thanks to recent advances in technology and software the instrumentation needed to perform 2D LC is broadly available to the analytical community in both industry and academia Indeed the technique can now be considered ready for application in R D as well as in QA and QC labs yet it is not widely known about outside academic laboratories and is rarely taught at the undergraduate level This book outlines the main principles and features of 2D LC including comprehensive and heart cutting modes method development and real world applications to enable modern analysts to start using this fascinating technique

The book offers an ideal starting point for those wishing to get into 2D LC and will also be of interest to more experienced scientists in the field

Directory of Graduate Research, 2001 Faculties publications and doctoral theses in departments or divisions of chemistry chemical engineering biochemistry and pharmaceutical and or medicinal chemistry at universities in the United States and Canada

Investigations Into Background Correction and Retention Time Alignment to Enhance Quantitative Chemometric Analysis of Comprehensive Two-dimensional Liquid Chromatography-diode Array Detection Data Robert Craig Allen, 2012 The focus of the projects presented here was to develop possible solutions to three issues commonly encountered during chemometric analysis of comprehensive two dimensional liquid chromatography diode array detector LCxLC DAD data The focus of the first project was to determine a means of performing background correction that removed two background ridges The methods of simply subtracting out a mean blank sample singular value decomposition based background correction SVD BC and asymmetrically weighted least squares AWLS were compared AWLS was found to be the only background correction technique to fully remove the ridges However AWLS was also found to attenuate the peak intensity by approximately 25% due to over fitting of the background at the lower wavelengths The focus of the second project was the investigation of five common interpolation strategies for the reconstruction of the sampled first dimension peak The interpolation strategy that best reproduced the original first dimension retention time was Gaussian fitting This was expected given that the simulated data set was generated using a Gaussian model for the peak shape An algorithm semi automated alignment method SAAM was then developed that allowed for each peak to be aligned independently of the other peaks in the data set SAAM was validated using both simulated and experimental data The simulated results indicated that SAAM produced percent recoveries close to 100% SAAM was also compared to iterative key set factor analysis alternating least squares IKSFA ALS for the analysis of phenytoin in a waste water treatment plant effluent SAAM produced a concentration of 26.3 ppb compared to 39.9 ppb from IKSFA ALS While these results are very different the result produced by SAAM is still within the experimental error of the reference 2D LC M S MS method 42.19 Finally SAAM was compared to two existing literature methods A mixture of simulated and experimental data sets was used to measure the accuracy and precision of the results SAAM was found to be impacted less by intra and inter sample retention time shifting then PARAFAC2 SAAM and shifted candecomp PARAFAC were found to produce very similar results However SAAM was found to experience some difficulty producing accurate and precise results with some of the experimental data sets

Basic Multidimensional Gas Chromatography, 2020-03-20 Basic Multidimensional Gas Chromatography is aimed at the next generation of multidimensional gas chromatography users who will require basic training in the fundamentals of both GC and GCxGC This book fills the current need for an inexpensive straightforward guidebook to get new users started It will help new users determine when to add or purchase a multidimensional system and teach them to optimize and maximize the capability of each system Readers will also learn to select specific modes for each portion of a multidimensional analysis This

ideal resource is a concise hard hitting text that provides the facts needed to get users up and running Provides a comprehensive and fundamental introduction to multidimensional gas chromatography Assists readers in determining when to add or purchase a multidimensional system Explains how a given system can be used to its maximum capacity and how users should choose specific modes for different portions of multidimensional analysis **Nutrition Abstracts and Reviews** ,2005 Advanced Techniques in Gas Chromatography-Mass Spectrometry (GC-MS-MS and GC-TOF-MS) for Environmental Chemistry ,2013-12-10 Gas chromatography mass spectrometry GC MS has been the technique of choice of analytical scientists for many years The latest developments in instrumentation including tandem mass spectrometry MS MS and time of flight TOF detectors have opened up and broadened the scope of environmental analytical chemistry This book summarizes the major advances and relevant applications of GC MS techniques over the last 10 years with chapters by leading authors in the field of environmental chemistry The authors are drawn from academia industry and government The book is organized in three main parts Part I covers applications of basic GC MS to solve environmental related problems Part II focuses on GC MS MS instrumentation for the analyses of a broad range of analysis in environmental samples pesticides persistent organic pollutants endocrine disruptors etc Part III covers the use of more advanced GC MS techniques using low and high resolution mass spectrometry for many applications related to the environment food and industry

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