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 Chiral Separations By Liquid Chromatography And Related Technologies

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Chiral Separations Wiley-VCH

From the contents: Chiral chromatographic separations based on ligand exchange (A. Kurganov). - Chiral separations using the macrocyclic antibiotics: a review (T.J. Ward, A.B. Farris III). - High-performance liquid chromatographic and capillary electrophoretic enantioseparation of plant growth regulators and related indole compounds using macrocyclic antibiotics as chiral selectors (Review) (F. Hui et al.). - Polysaccharide-based chiral stationary phases for high-performance liquid chromatographic enantioseparation (Review) (E. Yashima).

Chiral Separation Techniques Springer Science & Business Media

Enantiomeric separations are an essential component of pharmaceutical drug development, not only at the analytical scale, but also to separate usable quantities for further analysis. The field of asymmetric synthesis is also heavily dependent on chromatographic methods to separate and

quantitate the results of asymmetric transformations as well as characterize new ligands and catalysts. This dissertation focuses on the use of macrocyclic chiral stationary phases for use in high performance liquid chromatography as well as subcritical fluid chromatography to separate individual enantiomers of molecules of importance to the scientific community. Optimized separation conditions are provided for many of these important analytes, which will expedite the evaluation of their usefulness in a variety of applications. Particular emphasis is put on elucidating the mechanism of interaction between analyte and stationary phase. In chapters two and three, principle component analysis is applied to the chromatographic data to gain better understanding of the factors contributing to retention and enantioselectivity. It was shown that optimized separation conditions are also provided for newly synthesized isochromene and Tröger base derivatives using cyclodextrin and cyclofructan based chiral stationary phases. The fourth chapter provides separation conditions for a variety of novel synthetic biaryl atropisomers, which have the potential to serve as useful ligands in asymmetric transformations as well as possessing antibiotic/antimicrobial properties. Preparative scale separation conditions are also provided allowing for these important analytes to be prepared and evaluated in their enantiomerically pure

form. Insight into the mechanism of analyte retention is provided indicating that dipolarity/polarizability is the primary retentive interaction between substituted biaryls and derivatized cyclofructans. Chapter five provided a valuable comparison of commonly used chromatographic conditions for the separation of primary amines using cyclofructan based chiral stationary phases. The effect of various additives and polar modifiers was investigated and the results indicate that a combination of acidic and basic additives is necessary to obtain optimal separations. The advantages of individual chromatographic modes are also provided. Normal phase separations provided the greatest selectivities at the cost of longer analysis times while modified carbon dioxide mobile phases provided excellent peak profiles and short analysis times. Preparative scale separations are also provided using modified carbon dioxide mobile phases allowing for enantiopure compounds to be prepared in an environmentally friendly manner without the use of petroleum based solvents.

Recent Advances in Chiral Separations Springer Science & Business Media
 CHIRAL SELECTORS, CHIRAL SEPARATION, OXAMNIQUINE, CIPROFIBRATE, CYCLODEXTRINS AND THEIR ..., HEPARIN, METHOD OPTIMISATION, HIGH-PERFORMANCE LIQUID ..., CAPILLARY ZONE

ELECTROPHORESIS, WHELK-O 1 STATIONARY PHASE.

Chiral Separation Techniques CRC Press

Biological in vivo processes are stereochemically controlled and rate limited by proper selection of enantiomers. Wrong selection can have deleterious effects, therefore, more than 40% of drugs (over the retail counters and prescribed) are indeed chiral, and of these 25% are supplied as pure enantiomers. So chiral separation has remained interesting and still challenging task for oneself to develop the new, simple, reproducible and sensitive methods. This book focuses on the chiral separation of some important pharmaceuticals using two major approaches; one is pre-column derivatization with a chiral reagent followed by separation of resulting diastereomers known as 'indirect approach'. The other one is 'direct approach' which may use a chiral mobile phase additive (CMPA) or a chiral stationary phase (CSP) or the chiral selector is immobilized/impregnated with the stationary phase.

Chiral Analysis Wiley

The development of chiral liquid chromatography, facilitating the straightforward separation of enantiomers, was a significant advance in chromatography, leading to widespread application in analytical chemistry. Application in preparative chromatography has been less rapid, but with the development of single enantiomer pharmaceuticals its use is increasingly common in chemical synthesis at laboratory, pilot plant and even full production scale. Brings non-experts up to speed quickly and comprehensively, facilitating the rapid development of effective separations of enantiomeric mixtures on a range of process scales. Presents case studies drawn from within the pharmaceutical industry to clearly illustrate the utility and value of preparative scale enantioselective chromatography in chemical research, development and production. Key reference source and entry to the literature so the reader does not have to engage in expensive and time-consuming literature searching.

Chiral Separations Elsevier Science Limited

This chapter summarizes major developments in the field of liquid chromatographic separation of enantiomers. After a short historical overview, the materials and technologies used for analytical and preparative scale separation of enantiomers in high-performance liquid chromatography, nano liquid chromatography, simulated moving-bed chromatography, and supercritical fluid chromatography are briefly discussed. In the final part, some future trends in liquid chromatographic separation of enantiomers are overviewed.

Chiral Separation of Drugs and Related Compounds by High-performance Liquid Chromatography Elsevier

Modified Cyclodextrins for Chiral Separation offers a review of the latest advances in developing modified cyclodextrins as chiral selectors for various chromatographic and electromigration techniques. Over the years, many descriptions of chiral separation have appeared in academic journals and books, but most of them have been devoted to either the development of analytical methods and protocols or the summary of different chiral selectors, including cyclodextrins for chiral separation applications. This is in marked contrast to this volume which focuses on the research endeavors concerning the development of cyclodextrin derivatives specifically as either chiral mobile phases for capillary electrophoresis, or chiral stationary phases for various chromatographic techniques including gas chromatography, or high-performance liquid chromatography and supercritical fluid chromatography. The ongoing thread in this book is the synthesis of structurally-defined cyclodextrin derivatives and their applications in enantiomer separation by means of different analytical techniques. Modified Cyclodextrins for Chiral Separation is intended for those who are interested in expanding their knowledge of cyclodextrin chemistry and chiral separation, and in what cyclodextrin modification can be made to suit the needs of chiral selectors for different analytical techniques. It primarily focuses on the state-of-the-art cyclodextrin chemistry which is the basis for all chiral selectors used in these chiral separation techniques. Weihua Tang, PhD, is a professor at the Key Laboratory of Soft Chemistry and Functional Materials, Ministry of Education, Nanjing University of Science and Technology, China. Siu-Choon Ng, PhD, is a professor at the Division of Chemical and Biomedical Engineering, School of Chemical and Biomedical Engineering, Nanyang Technological University, Singapore. Dongping Sun, PhD, is a professor at the Key Laboratory of Soft Chemistry and Functional Materials, Ministry of Education, Nanjing University of Science and Technology, China.

Chiral Analysis John Wiley & Sons

Prominent experts from around the world detail the chromatographic and electroseparation techniques they have developed for chiral separations on an analytical scale. Described in step-by-

step detail to ensure successful experimental results, the procedures are presented as either general methods or as specific applications to substance classes and special compounds, with emphasis on high performance liquid chromatography and capillary electrophoresis techniques, but also including thin layer chromatographic, gas chromatographic, supercritical fluid chromatographic as well as recent electrochromatographic techniques.

New Bio-analytical Separations Utilizing Chiral Mobile Phase Additives in Thin Layer Chromatography and Chiral Stationary Phases in High Performance Liquid Chromatography Springer Science & Business Media

"The problem addressed by this dissertation is the separation of optical isomers in commercial as well as biological samples. The chromatographic separation of enantiomers is an important and rapidly developing field of study. Chiral separations of pharmaceutical compounds and important organic intermediates in high performance liquid chromatography (HPLC) and thin layer chromatography (TLC) were achieved. Two methods were employed for the direct liquid chromatographic resolution of chiral analytes: chiral stationary phases (CSPs) and chiral mobile phase additives (CMAs). Native and derivatized [beta]-cyclodextrins ([beta]-CD) were used as chiral stationary phases in reverse phase and normal phase HPLC, respectively. This study marked the first use of derivatized [beta]-CDs for chiral separations in normal phase media. N-carbobenzoxy-glycyl-L-proline and (1R)-(-)-ammonium-10-camphorsulfonate were utilized as CMAs in normal phase TLC for the resolution of several aromatic amino alcohols. Maltosyl-[beta]-CD and hydroxypropyl-[beta]-CD were employed as CMAs in reverse phase TLC. A study was conducted with hydroxypropyl-[beta]-CD to determine how the degree of substitution of a derivatized CD could effect development time, the viscosity of the solution and the enantioselectivity. In addition, studies were initiated to determine the presence of trace levels of D-amino acids in: amniotic fluid, blood serum and urine. The blood and urine of healthy young adults were analyzed and found to contain trace to percent levels of D-amino acids. The human amniotic fluid samples did not have detectable levels of D-amino acids"--Abstract, page iv.

Chiral Recognition in Separation Methods CRC Press

Chiral Analysis: Advances in Spectroscopy, Chromatography and Emerging Methods, Second Edition covers an important area of analytical chemistry of relevance to a wide variety of scientific professionals, including chemistry graduate students, analytical chemists, organic chemists, professionals in the pharmaceutical industry, and others with an interest in chirality and chiral analysis. This thoroughly revised second edition covers several new, important areas of chiral analysis that have emerged since the first edition. Three of the new methods provide higher sensitivity than can be realized with the current methods and are expected to become mainstream applications: cavity based methods offer vastly higher sensitivity than conventional polarimetric methods, microwave chiral detection provides unsurpassed sensitivity for identifying diastereomers, and the rotating electric field method offers a competing new approach for the separation of enantiomers. Another topic, chirality in extraterrestrial life, has not been discussed in any other book and is important for understanding the origin of life. Offers the only book to cover both spectroscopic and separation methods in a single volume. Provides an up-to-date and detailed review of the various techniques available, including new techniques that have emerged since the first edition. Includes contributions from a range of leading experts in the field, now edited by award-winning chirality researcher Prasad Polavarapu.

Studies of Enhanced-fluidity Liquids for Chiral Separations John Wiley & Sons

Both analytical and preparative-scale enantioseparation techniques are covered in a down-to-earth practical way. The most important aspects of design, economics and safety are considered with emphasis on current European and North American legislation. In addition, the theory of chiral separation is covered in sufficient detail to guide the practising chromatographer interested in developing new techniques. A team of experts from academic and industrial laboratories throughout the world have compiled their findings and experience to make this book an exceptionally timely and unique contribution to the field.

Chiral Separation of Pharmaceutical Compounds by High Performance Liquid Chromatography John Wiley & Sons

Discusses chiral separations and offers guidance for selecting the optimum method for desired results. Chiral separations represent the most intriguing and, by some measures, most difficult separations of chemical compounds. This book provides researchers and students an understanding of chiral separations and offers a convenient route to selecting the best separation method, saving considerable time and cost in product development. Considering chiral separations

in the biotechnological and pharmaceutical industries, as well as for food applications, Dr. Ahuja provides insights into a broad range of topics. Opening with a broad overview of chiral separations, regulatory considerations in drug product development, and basic issues in method development, the book covers a variety of modern methods such as gas chromatography, high performance liquid chromatography, supercritical fluid chromatography, and capillary electrophoresis. Deals with the impact of chirality on the biological activity of small and large molecules. Provides detailed information on useful chiral stationary phases (CSPs) for HPLC. Includes handy information on selection of an appropriate CSP, including mechanistic studies. Offers strategies for fast method development with HPLC, SFC, and CE. Discusses preparatory methods utilized in the pharmaceutical industry. With in-depth discussions of the current state of the field as well as suggestions to assist future developments, *Chiral Separation Methods for Pharmaceutical and Biotechnological Products* is an essential text for laboratory investigators, managers, and regulators who are involved in chiral separations in the pharmaceutical industry, as well as students preparing for careers in these fields.

Attempt to Improve Resolution and Efficiency of Reversed-phase Chiral Separation Using High-performance Liquid Chromatography John Wiley & Sons

Though many separation processes are available for use in today's analytical laboratory, chromatographic methods are the most widely used. The applications of chromatography have grown explosively in the last four decades, owing to the development of new techniques and to the expanding need of scientists for better methods of separating complex mixtures. With its comprehensive, unified approach, this book will greatly assist the novice in need of a reference to chromatographic techniques, as well as the specialist suddenly faced with the need to switch from one technique to another.

Preparative Enantioselective Chromatography Springer Science & Business Media

This research explores the application of a new technique, termed electrochemically modulated liquid chromatography (EMLC), to the chiral separations of pharmaceutical compounds. The introduction section provides a literature review of the technique and its applications, as well as a brief overview of the research described in each of the next two chapters. Chapter 2 investigates the EMLC-based enantiomeric separation of a group of chiral benzodiazepines with [beta]-cyclodextrin as a chiral mobile phase additive. Chapter 3 demonstrates the effects of several experimental parameters on the separation efficiency of drug enantiomers. The author concludes with a general summary and possible directions for future studies. Chapters 2 and 3 are processed separately.

Chiral Separation Methods for Pharmaceutical and Biotechnological Products Chiral Separation Techniques

While working as a chromatographer in the pharmaceutical industry, it became apparent to the editor that there was a pressing need for a comprehensive reference text for analysts working on the resolution of enantiomers by liquid chromatography (LC). This need arises from the fact that, whereas previously it was very difficult to determine enantiomers by direct means, there is now a wide choice of direct LC methods. At the same time, regulatory authorities have been changing their attitudes towards the administration of pharmaceuticals as racemates, partly because it is now possible to study the individual enantiomers. Clearly this abundance of new information needs to be rationalized. More importantly, the chiral LC systems which are commercially available or readily accessible to the practising chromatographer needed to be reviewed and, to a much greater extent than in existing reviews or books, discussed in terms of their practical application. Accordingly this book is very much orientated towards the practical aspects of these commercially available and readily accessible chiral LC systems. To this end, it is written for practising chromatographers by a team of practising, experienced chromatographers who have spent many years tackling the problems presented by resolving enantiomers by LC. The practical aspects of common chiral LC systems cannot be fully understood if discussed in isolation.

Liquid Chromatography Elsevier

HPLC and CE: Principles and Practice presents the latest information on the most powerful separation techniques available: high-performance liquid chromatography (HPLC) and capillary electrophoresis (CE). Fundamental theory, instrumentation, modes of operation, and optimization of separations are presented in a concise, non-technical style to help the user in choosing the appropriate technique quickly and accurately. Well-illustrated and containing convenient end-of-chapter summaries of the major concepts, the book provides in-depth coverage of troubleshooting, improvement of resolution, data manipulation, selectivity, and sensitivity. Graduate

students, technicians, and researchers who must use separations with little or no background in analytical chemistry can overcome separation anxiety and get started in obtaining the best possible separations in minimal time. The book will also be useful to analytical chemists who need a better understanding of theory and processes. Fully up-to-date information on both HPLC and CE includes troubleshooting and comparisons of the two techniques Applicable to a wide variety of separation problems Covers basic concepts governing any separation as well as instrumentation and how to use it Helps the user to obtain optimal resolution in minimal time Contains information on special procedures such as chiral separations, affinity chromatography, and sample preparation Includes information on upcoming trends such as miniaturization Major concepts in each chapter are organized to allow access to information easily and quickly Contains practical bibliography for accessing the literature

Chiral Separations by Capillary Electrophoresis Springer Science & Business Media

While changes in the E_{app} to the stationary phase play a major role in the alteration of efficiency and elution order of the enantiomers, results also show that the enantioselectivity and retention are influenced by the identity and the concentration of the organic additive, and by the pH and flow rate of the mobile phase. The observations are discussed in terms of chemical structure and retention relationships.

Principles and Practice of Modern Chromatographic Methods Springer Science & Business Media

What drives a scientist to edit a book on a specific scientific subject such as chiral mechanisms in separation methods? Until December 2005, the journal *Analytical Chemistry* of the American Chemical Society (Washington, DC) had an A-page section that was dedicated to simple and clear presentations of the most recent techniques or the state of the art in a particular field or topic. The "A-page" section was prepared for a broad audience of chemists including industrial professionals, students as well as academics looking for information outside their field of expertise. Daniel W. Armstrong, one of the editors of this journal and a twenty-year+ long friend, invited me to present my view on chiral recognition mechanisms in a simple and clear way in an "A-page" article. In 2006, the "A-page" section was maintained as the first articles at the beginning of each first bi-monthly issue but the pagination was no longer page distinguished from the regular research articles published by the journal. During the time between the invitation and the submission, the A-page section was integrated into the rest of the journal and the article appeared as (2006) *Anal Chem* (78):2093-2099.

Chiral Chromatography Wiley-VCH

A single source of authoritative information on all aspects of the practice of modern liquid chromatography suitable for advanced students and professionals working in a laboratory or managerial capacity Chapters written by authoritative and visionary experts in the field provide an overview and focused treatment of a single topic Comprehensive coverage of modern liquid chromatography from theory, to methods, to selected applications Thorough selected references and tables with commonly used data to facilitate research, practical work, comparison of results,

and decision making Extensive original tables and figures, placing recent research developments into a general context Worked examples, intuitive explanations, and clear figures reinforce learning

High Performance Liquid Chromatography & Capillary Electrophoresis John Wiley & Sons

The design of chiral separations in liquid chromatography (LC) and capillary electrophoresis (CE) involves the selection of chiral selectors and eluent parameters, often on a purely empirical basis. It would be desirable if rapid screening methods could be designed to rationalise the choice of these chiral selectors. With reference to the use of cyclodextrin (CD) derivatives as chiral selectors, nuclear magnetic resonance spectroscopy (NMR) can play an important role in screening the extent of interactions with chiral solutes, and in probing the nature of the stereoselective interactions involved. Data from high-field NMR on drugs and their derivatives have been explored for screening a number of potential chiral recognition agents, as an aid to the rational design of chiral separations by LC and CE, based on cyclodextrins (alpha, beta, gamma, hydroxypropyl-beta and hydroxyethyl-beta) bonded to silica (LC) and in free solution (CE). In this investigation high-field FT-NMR is used to examine the interaction mechanism between these cyclodextrins and the calcium channel blocker Amlodipine together with a series of its structural analogues. Enantiomeric interactions between the cyclodextrins and Amlodipine are explored using ROESY (rotating frame nuclear Overhauser effect spectroscopy) in order to determine the inclusion mechanism involved.....

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