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A Review of: Pulsed Electrochemical Detection in High-Performance Liquid Chromatography

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THE BOOK CORNER

TIME-OF-FLIGHT MASS SPECTROMETRY, INSTRUMENTATION AND APPLICATIONS IN BIOLOGICAL RESEARCH, R. J. Cotter, American Chemical Society Professional Reference Books, American Chemical Society, Washington, DC, 20036, USA, 1997, 320 pp., \$119.95.

One of the strengths of this book is its identification and elucidation of the basic principles of time-of-flight mass spectrometry (TOF-MS). There are also numerous topics covering instrumentation, applications, and even a brief history of the TOF spectrometer, but discussions of advanced topics are well supported by thorough examination of underlying principles. In fact, this monograph could serve as a text or reference on different levels, depending upon the readers' needs and desires. There are many figures, spectra, and references, and detailed derivations of equations are presented in highlighted boxes for the hardy.

Chapters 1-3 discuss considerations fundamental to all types of TOF instruments, including time, space, and kinetic energy distributions of ions, ion focusing, and ion fragmentation. Chapter 1 is an interesting, brief history of the evolution of TOF-MS. Much of the content of Chapter 2 has been published previously by Professor Cotter, but it is worthwhile to include these integral topics as part of this comprehensive book. Chapter 3 presents a lucid and readable discussion of energy focusing devices, primarily reflectrons.

Chapters 4-6 introduce a variety of approaches utilizing TOF-MS, distinguished primarily by the desorption/ionization method used. Some discussion of the relevant desorption/ionization mechanisms are included, as well as distinguishing features of each approach.

Chapters 7-9 offer a somewhat more specialized, but still quite broadly applicable presentations of ion extraction strategies, including delayed extraction, orthogonal extraction, and ion storage. Further elaboration on ion trap-TOF instruments would seem justified, given current trends.

Chapter 8 is an excellent treatment of ion fragmentation and, more specifically, post-source decay. A brief survey of tandem and hybrid TOF instruments is included in Chapter 9.

Chapters 10-11 describe the applications of TOF-MS to the characterization of biopolymers. While there are no experimental details (these are not within the scope of this book), there are lots of references to enable further investigation of the many techniques described. Chapter 10 deals with protein and glycoprotein applications, providing a survey of the strategies employed. Absent from this discussion are the chemical and enzymatic treatments of proteins within polyacrylamide gels, and attempts to analyze proteins directly from ultra-thin gels. Chapter 11 is a rarely found discourse of the strategies and difficulties associated with the analysis of oligonucleotides and DNA by TOF-MS. An especially illuminating feature of this chapter is the inclusion of hybridization strategies in genomic analysis.

This book is highly recommended. The emphasis on basic principles ensures that much of the material included will be relevant as TOF-MS evolves, and will help the reader evolve with the technique.

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HPLC METHODS FOR PHARMACEUTICAL ANALYSIS, G. Lunn, N. R. Schmuff, John Wiley & Sons, Inc., New York, 1997, 1609 pp., \$150.00.

The book is a collection of procedures for the analysis of a number of pharmaceuticals using high-performance liquid chromatography (HPLC). Procedures for approximately 150 compounds are listed. The drugs were selected according to the authors, "based on surveys of the top 200 drugs by number of prescriptions filled and the top 100 drugs by dollar sales, we selected the most commonly used drugs in the United States." I believe that is a fair way to do it.

Each monograph comprises two sections: Procedures and Annotated Bibliography. The first section presents detailed procedures which should enable anyone to reproduce the analyses. The second section lists other relevant papers, but does not give any experimental details. However, some key words are given to indicate key features of the procedure that are not referred to in the title. The intent of this section is to allow readers to determine, rapidly, if a paper is relevant and worth looking up in the library.

The detailed procedures given in the first section of each monograph normally contain the following sections in addition to name, molecular and structural formulae, molecular weight, and CAS registry. Of course, not all papers give full details, so some sections may be missing: Reference, Matrix, Sample Preparation, Guard Column, Column, Mobile Phase, Flow Rate, Injection Volume, Retention Time, Detector, Internal Standard, Limit of Detection, Limit of Quantitation, Drugs That Are Extracted Under These Conditions, Drugs That Are Chromatographed Simultaneously Under These Conditions, Drugs That Are Also Chromatographed Under These Conditions, Drugs That Are Non-Interfering, Drugs That Are Interfering, and Key Words.

The matrices mentioned in the procedures are: bile, blood (plasma, serum,

whole blood), bulk, CSF, dialysate, formulations (capsules, injections, tablets, creams, ointment, etc.), microsomal incubations, milk, perfusate, reaction mixtures, saliva, solutions, tissue (muscle, kidney, liver, heart, spleen, brain, etc.), and urine.

The procedures are listed in the alphabetical order of the compounds. For some, such as acetaminophen, there are 26 pages containing 37 different procedures; for others, only one or two procedures are given. The authors did a commendable job on designing the monographs, selecting the compounds, and the thorough search of the literature. The result is a listing of procedures without any input from the authors as to the quality of each method. For example, was the peak of interest well resolved?, was the peak broad, skewed or sharp? For acetaminophen, which of the 37 methods listed is the recommended HPLC procedure to follow?! Also, there is no mention of reproducibility or statistical data as to the quality of the method. So, the reader is left on his own to decide (or guess) what procedure to follow.

In spite of these omissions the book is a massive work, over 1600 pages for less than 10 cents a page, the cheapest I have seen in a long time. It is worth the price for those involved in the analysis of pharmaceuticals.

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Editor,
The Book Corner

HPLC PRACTICAL AND INDUSTRIAL APPLICATIONS, J Swadesh, Ed., CRC Press, Boca Raton, FL, 1996, 358 pp., \$89.95.

The success of a technique is indicated by the number of books, manuscripts written about it, and its uses. Although many publications have been written about HPLC, this journal received three books that deal with high performance liquid chromatography:

- 1) HPLC Practical and Industrial Applications, edited by Joel Swadesh;
- 2) HPLC Columns, Theory, Technology and Practice by Uwe D. Neue;
and
- 3) Pulsed Electrochemical Detection in High Performance Liquid Chromatography by William R. LaCourse.

Although one wonders why we need more books about HPLC, it is refreshing to see that more books are written about it, using different approaches. Some of the material might have been written about in another of the comprehensive texts already available to the reader, but it does not cause any harm to have it repeated in a different format. The more the better! Each of the above three books will be reviewed separately.

According to the editor, "The organization of the present work is somewhat unconventional in that it is structured to facilitate problem-solving. Books on chromatography are conventionally divided into theory, instrumentation, and practice, or into isocratic vs. gradient techniques, or by class of analyte. In the present work, information is clustered around certain topics in a manner to aid rapid problem-solving."

The purpose of this book, as stated in the Preface, "is to examine analytical HPLC as it is actually used in industry. Rather than focus on the technique issues alone, the book acknowledges that technical issues are inseparably intertwined with non-technical issues."

The editor also states that, "The technical aspects of analytical HPLC are the principal focus of the present work. The goal is to impart the generalist's breadth with the specialist's depth."

Accordingly, this book is organized to facilitate rapid absorption of a particular area of chromatography. The first chapter is a general chapter on instrumentation, theory, and laboratory operations, designed for the non-specialist unexpectedly drafted into analytical chemistry. A brief survey of absorbance, fluorescence, and refractive index detectors is presented.

Pumps and columns are also described. Detailed information on specialty detectors, such as electrochemical, viscosimetric, and light-scattering detectors, is presented in later chapters in association with those chromatographic modes with which they are commonly used.

The second chapter is designed for the traditional analytical chemist who is transferred into the manufacturing environment. It covers process sampling and analysis. The third chapter describes process chromatography.

The remaining four chapters are on specialities within separations technology, i.e., reversed phase chromatography, ion exchange chromatography, gel permeation chromatography, and capillary electrophoresis. Each of these chapters includes an introductory section to outline the key features of the technique, a thorough bibliography, and list of precedents, and detailed examples of one or more applications, presented from the viewpoint of an industrial scientist.

Specialty detectors are also described in these chapters. The chapter on ion exchange discusses electrochemical detectors, and the chapter on gel permeation chromatography describes light-scattering and viscosimetric detectors.

The book is well written by scientists from industry who faced different analytical problems and how to solve them. In general, the text is focused on troubleshooting and a problem solving guide for those who use HPLC in their daily work.

The more than 100 figures and 1000 references will allow for easy access to certain specific problems. This text is a welcome addition to the HPLC library.

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7. High Performance Capillary Electrophoresis: An Overview, K. Benedek and A. Guttman, (305).

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The Book Corner

HPLC COLUMNS, THEORY, TECHNOLOGY, AND PRACTICE, U. D. Neue, Wiley-VCH Publishers, New York, 1997, 393 pp., \$89.95.

As the title implies, this book, unlike others dealing with HPLC, is dedicated totally to HPLC columns. Although many chapters have been written in the past 25 years about columns and column packings, no book was published dealing with the heart of the HPLC system, the column. Therefore, this book, which discusses all aspects of HPLC columns in depth, is a timely addition to the many books published about HPLC.

The author writes: "One of the first steps in writing a book is to define the audience for which it is written. I decided to address the text to the HPLC practitioner working in the laboratory. Therefore, the book is largely a practical guide, but it also contains sufficient theoretical background information to give an in-depth understanding of the topics covered. Thus, it should be useful for both the novice and the experienced user of chromatography."

"Another decision that needs to be made early is which subjects should be covered and what should be omitted. Since the book is intended for the HPLC user, I limited the discussion of packings and columns largely to those that are commercially available. At the same time, I did not include the lengthy tables of commercially available products found in some other books. In my experience, these tables are far from complete and full of erroneous information." I agree with the author's statement.

The book is divided into 17 chapters. Chapter 1 is an introduction which deals with history, separation mechanisms, scope of the book and asks the question: What is HPLC? Theory of chromatography is discussed in detail and in a clear manner in Chapter 2. The next four chapters deal with column design, packing, column packing and testing, and column chemistry. These are comprehensive discussions which give the reader an excellent overview. Topics

such as particles' physical properties, e.g., particle size, shape, porosity, and strength are discussed in detail in Chapter 4. Bonding, monomeric vs. polymeric and other interesting topics are discussed in Chapter 6. Column selection, which includes topics such as polymer, protein, peptide, nucleic acids, and carbohydrate analysis is discussed in Chapter 7.

Chapters 8-13 are devoted to different HPLC modes of separation such as size-exclusion, normal-phase, reversed-phase, hydrophilic interaction, ion-exchange and hydrophobic interaction chromatography. The largest portion of the discussion is devoted, justifiably, to reversed-phase analyses. Column coupling, switching, and other topics are elaborated upon in Chapter 14, while Chapter 15 deals with all aspects of preparative chromatography.

The important topic, from the analyst's point of view, methods development is discussed in detail by Dr. El Fallah. The book ends with a nice and useful discussion of column maintenance. This is a must book to have. It is clearly presented, well organized, and referenced. I highly recommend it.

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The Book Corner

PULSED ELECTROCHEMICAL DETECTION IN HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY, W. R. LaCourse, John Wiley & Sons, Inc., New York, 1997, 324 pp., \$59.95.

This book is another welcome addition to the HPLC library. This volume is another one of the Techniques in Analytical Chemistry Series. Previously, we reviewed another volume of this series, *Capillary Electrophoresis*, by D. Baker, which we liked very much as a basic book for the beginner. If the present text is as good as the other one, then we will be watching for the next volume in this series.

“The popularity of pulsed electrochemical detection (PED) has steadily increased over the last decade for the sensitive and reliable detection of many polar aliphatic compounds in aqueous samples, following their separations by high-performance liquid chromatography (HPLC). PED is based on multi-step potential waveforms applied at solid electrodes to alternate the operations of anodic detection with oxidative cleaning and reductive reactivation of the electrode surfaces.

Compounds detected by these multi-step waveforms, at gold and platinum electrodes, include all alcohols, alditols, and carbohydrates, as well as many organic amines and sulfur compounds. The development of HPLC-PED represents a successful marriage of two analytical technologies,” according to the author, and we agree!

The book is divided into three major parts: background material necessary for a more thorough understanding of the principles and relevance of PED; an in-depth discussion of PED using voltammetry and other electroanalytical techniques and presenting the advantages, applicability, and optimization of all existing PED waveforms; and practical aspects of HPLC-PED, including a summary of the major applications and a look at future developments in the technique. Appendices include a pulsed voltammetry (PV) program specifically written to optimize pulsed amperometric detection (PAD) waveforms and all the known applications, categorized and listed in tabular form.

The author is correct in stating: "Because the principles of HPLC have been summarized in numerous monographs, the author has made the appropriate choice to emphasize the electrochemical principles on which PED technology is based." I am glad to see that Dr. LaCourse concentrated his emphasis on PED and not on discussing the theory of chromatography. Many authors, however, fall into this trap of discussing principles either poorly or not in sufficient detail at the beginning of their books. It is preferable to devote the book to the topic of discussion. The book contains nine chapters. The discussion is readable and easy to follow. Dr. LaCourse presents the material in such a way that the novice, as well as the experienced, will benefit from reading it. The book is well illustrated and referenced.

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