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Book Reviews

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BOOK REVIEWS

Carbon, Nitrogen, and Sulfur Pollutants and Their Determination in Air and Water by Jerome Greyson (Metropolitan State College, Denver; J&JG Associates, Conifer, CO, USA), 376 pages, Marcel Dekker Inc., New York (1990), ISBN 0-8247-8235-6, US\$ 119.5.

C, N, and S are involved (along with oxygen and phosphorus) in the main biogeochemical cycles which make the Earth functioning like it does. Getting reliable analytical data of C, N, and S compounds is, therefore, a main goal for both environmental scientists and industrial waste monitoring chemists.

The book is structured into four parts. **Part I** contains an overview on the beginning and evolution of pollution-related problems as understood in the present, stressing the important role that C, N, and S play in this context. Furthermore, the natural cycles of these elements, their relationships with the biosphere, and their anthropogenic interferences, are reviewed.

Part II deals with the occurrence and behaviour of the three elements as pollutants. After a description of chemical properties of their most important compounds and their biochemical roles, the author turns to discuss their occurrence in water and air. Properties of both air and water as pollution receptors are considered, as well as some notions of physico-chemical equilibria of pollutants in these media.

Part III contains the main subject of the book: a compilation of all available, recommended methods for the determination of C, N, and S compounds in environmental samples. This part is structured into six chapters, each devoted to one family of analytical techniques: physical methods (Spectroscopy, Turbidimetry and Nephelometry, Luminescence and Fluorescence, Mass Spectrometry), electrochemical methods (Potentiometry, Conductometry, Kinetics), chromatography (GC, LC, TLC), chemical methods (Gravimetry, Volumetry, Gas Detection Tubes, Combustion, Low Temperature Chemical Oxidation, Continuous Flow Analyzers), and bioanalytical methods (Enzyme and Immunochemical Analyses). In all cases the description of the instrumental setups and applications is preceded by a, sometimes too naive, presentation of the theoretical principles, (physical/chemical equations included). At the beginning of this part some notions about sampling, sampling statistics, and analytical errors are given.

Finally, in **Part IV** the author briefly (only two pages) comments on commercial equipments.

This book fills a gap between the massive number of journal articles on specific applications of the analysis of these elements in the environment and the numerous treatises on general analytical organic chemistry. The intended readers are all those nonanalytical chemists and engineers who are engaged in pollution monitoring

activities. It is also a valuable reading for upper-level undergraduates and graduate students in such disciplines.

This monograph is said to be a *practical reference* book. Taken from this point of view one misses a more practical approach because some important issues are ruled out or too shortly explained, e.g. sampling techniques, sample handling, inter-comparison of different methods, etc. Likewise, a more extensive and clear description of applications would be appreciated, so that one could get informed parting from his actual request. Thus, this comprehensive monograph would be even more environmentally useful.

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Chromatography/Fourier Transform Infrared Spectroscopy and Its Applications by Robert White (Dept. Chemistry, The University of Oklahoma, Norman, Oklahoma, USA), 328 pp., Marcel Dekker Inc, New York (1990), ISBN 0-8247-8191-0, US\$ 119.50.

This book is mainly addressed to analysts and researchers interested in a broad introduction to chromatography/FTIR. However, a good presentation of structural elucidation by FTIR is also included.

The book is structured around four main topics: general characteristics and problems of chromatography/FTIR interfaces (chapter 1), instrumental aspects of interfaces for GC, HPLC and TLC separations (chapters 2–4), identification and structural elucidation by FTIR (chapter 5) and practical examples of chromatography/FTIR (chapter 6).

In an introductory chapter the author describes the general characteristics of chromatographic analytical methods and FTIR in a very basic way. In a second part, he studies the FTIR interface optimization parameters and the chromatographic FTIR data generation.

Chapters 2 to 4 describe, from an historical perspective, the GC, HPLC and TLC interfaces for FTIR. Instrumental aspects are well covered including the interface basis, constructive problems, performance and a brief comparison among them. These chapters also provide a good survey of references to those interested on interface constructive details.

Chapter 5 is a very interesting chapter entitled Structure Elucidation Methods. First, the author describes searching algorithms for IR spectra libraries, routinely used today, making a healthy emphasis on mismatch sources. Finally, progress on new strategies like pattern recognition techniques or expert systems are well described.

Chapter 6 illustrates the application of chromatography FTIR to the analysis of complex mixtures. These examples show the advantages and limitations of this

technique, taking into account sample preparation, sensitivity requirements and analysis time considerations.

Briefly, this book covers basic techniques and principles, current applications and future trends of chromatography/FTIR analytical methods, including structural identification tools.

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Modern NMR Techniques and Their Application in Chemistry. Edited by Alexander I. Popov and Klaas Hallenga, 680 pp., Marcel Dekker Inc., New York (1991), ISBN 0-8247-8332-8, US\$ 162.00.

By looking at the title of this book a simple question arises: is there a need for another book on NMR? My answer is yes, because the development of NMR spectroscopy is so fast that only specialists are able to follow it through research papers and most of the actual NMR books are also written for NMR specialists. This book is addressed to chemists or biochemists which want to catch up the evolution of this field, from both the theoretical and recent application perspectives.

The book is structured in five parts: NMR theory in liquid and solid state (chapters 1–4), chemical quantitative analysis by NMR (chapter 5), structural elucidation by NMR in organic and inorganic chemistry (chapters 6–7), dynamic process studies by NMR (chapters 8–9) and two dimensional studies of biopolymers (chapter 10).

Chapters 1 to 4 provide a good introduction to Fourier Transform NMR and explain the fundamentals about 1D and 2D NMR. 2D experiments are discussed in a rigorous way through the product operation formalism and the author introduces the concepts of coherence transfer pathway and phase cycling selection. The last two chapters discuss solid state NMR, including quadrupolar nucleus, as well as the theoretical and practical aspects.

Chapter 5 is an introduction to the quantitative analysis world. It contains a good explanation about the basic requirements that an NMR experiment must fulfill in this respect and contains many methodological suggestions. After reading this chapter, one may wonder why NMR is not more widely used for quantitative analyses of few component mixtures?

Chapters 6 and 7 cover the classical use of NMR; that is structure elucidation, especially in the case of organic compounds. Basic correlation and editing experiments are covered through a few examples but without experimental details.

Chapters 8 and 9 present the possibilities of determining physicochemical properties of organic compounds or chemical reactions by NMR. The authors focus the work on chemical equilibria, kinetics and exchange processes, providing many reference data and examples.

In chapter 10 are presented the theoretical basis and some recent applications of

2D-NMR experiments for the elucidation of the 3D shape of biomacromolecules (especially small proteins) in solution. It intends to provide an overview about biochemistry oriented 2D-NMR experiments with their possibilities, limitations and practical considerations.

In summary, this book represents an introduction to NMR for non-specialists but without leaving aside the theoretical basis that are explained in a correct quantum formalism that can be followed with little effort. It is especially useful to people interested in solid state, quantitative analysis, physicochemistry and biochemistry, reporting many methodological details and providing a good reference source. However, it can be of little interest to people concerned with the classical structure identification problems.

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Analysis of Seawater, by T. R. Crompton (Northwest Water Authority, Warrington, UK), 423 pages, Butterworths Scientific Ltd., Guildford GU2 5BH, UK (1989). ISBN 0-407-01610-4, £85.

This book provides a comprehensive review of methods for the determination of organic and inorganic substances in seawater.

Chapter 1 discusses a general and very important aspect of seawater analysis, namely sampling, that may invalidate final results no matter how sophisticated the analytical procedure will be. Chapter 2 discusses the determination of anions with particular reference to the interfering effects of the sample matrix. Dissolved gases in seawater are of interest in certain contexts and their determination is discussed in Chapter 3.

Chapter 4 covers extensively the application of new techniques, including both preconcentration and analysis, for the determination of trace metals in seawater. In the first part of the chapter elements are discussed singly in alphabetical order, then as groups of elements because the newer techniques often cover ranges of elements. The monitoring of radioactive elements is covered separately in Chapter 5.

A wide range of biogenic and anthropogenic organic compounds are discussed in detail in Chapters 6 to 8. Organometallics, many of these being produced by biologically induced metal methylation processes occurring in sediments and fish tissues are also included. Sampling techniques of the different marine compartments (e.g. the particulate and dissolved fractions) as well as sample preparation prior to analysis for organics are also considered. Finally, in Chapter 9, is reported the determination of oxygen demand parameters in seawater. Amongst others these include total, dissolved and volatile organic carbon and total inorganic carbon. The determination of other non-metallic elements, such as organic nitrogen and phosphorus, iodine and sulphur, is also discussed in this chapter.

The book discusses the advantages and disadvantages of the various techniques

available and provides almost 2000 references (755 on metals) on all aspects of the analysis of seawater constituents. Unfortunately, during the time elapsed between the literature up-dating (1985–6) and the publication date (1989), some important developments have occurred in the field which have not been possible to include. A few examples are the most recent extraction and preconcentration techniques for the determination of colloidal-dissolved organic constituents (e.g. "in situ" large volume sampling), the high temperature oxidation technique for DOC determination, reported by Sugimura and Suzuki in 1988, or the new analytical approach for the determination of individual components of PCB mixtures.

However, despite these small limitations this is a reference book, which will be invaluable to analysts, researchers and all those concerned with the scientific aspects and regulatory requirements for the preservation of the oceans.

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Mass Spectrometry, by E. Constantin and A. Schnell (Institute of Chemistry, University of Strasbourg, FR) 184 pages. Ellis Horwood Limited, England (1990). ISBN 0-13-555525-6. £39.95.

This is a short 18 chapter book that attempts to condense in about 180 pages most of the fundamental facts of mass spectrometry, including methods of ionization, mass analysis, sample introduction, data display, recording and processing as well as types of ions, fragmentation mechanisms, ion-molecule reactions, ion clusters, and general theory of mass spectra interpretation. The determination of nominal and exact mass, isotope analysis as well as some applications and recent developments are also discussed. Thus, coverage is extensive but necessarily limited in scope as the authors acknowledge in their own preface, so that treatment of the various topics is restricted to the essentials without too much room for detailed descriptions. Nevertheless, this type of book should be very useful to students and to readers wishing to obtain overall information about the theory and practice of mass spectrometry. Unfortunately, the book was written and first published in French in 1986 and translated in its present form in 1990 so that its most serious shortcoming would be the restricted coverage of the latest developments and applications.

In this regard a useful list of updated references has been added to the last chapter on recent developments together with an accompanying very short review section on the present state of the art. However, the latter would certainly merit a more extensive treatment in view of the giant leap forward experienced by mass spectrometric techniques in the past 3–4 years. In any case, the authors have managed to provide in an integrated volume a substantial amount of practical details and information on basic concepts with examples of their application in real life situations which, though mostly well known to the expert, are not easy to find in other books. In this late regard one must agree with the authors in that given the present state of

development of chemical instrument analysis, the teaching of mass spectrometry is still very much restricted or even absent in academic curricula and books such as this one can still serve a useful purpose.

Briefly, anybody interested in an overall description of the routine mass spectrometric methods and techniques available would benefit from this book but should not expect too much updated information on the latest ionization methods such as thermospray, electrospray, continuous flow FAB, matrix assisted laser desorption and the latest applications to the study of large biopolymers in the fields of molecular biology, biotechnology and biomedical sciences.

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Selective Sample Handling and Detection in High Performance Liquid Chromatography (Part B), Edited by K. Zech (Byk Gulden Pharmaceuticals, Konstanz, G.) and R.W. Frei (Free University, Amsterdam, NL). *Journal of Chromatography Library*, Vol. 39B, 384 pages, Elsevier, Amsterdam, The Netherlands, (1989). ISBN 0-444-88327-4. US\$ 129.25.

This book develops the idea emphasized in the first part of this volume that sample handling, separation, and detection in high performance liquid chromatography (HPLC) are integral, interrelated components of the analytical process rather than independent disciplines. The text successfully demonstrates that optimization of sample preparation may simplify detection (or be less demanding of the separation process) and vice versa.

The opening chapter examines the use of chelating silica supports for both preconcentration (liquid-solid extraction) and HPLC. The next chapter is an excellent, up-dated review of sample handling in ion chromatography that clearly highlights some of the unique procedures used in this technique with respect to sample collection, dissolution, cleanup, and avoidance of contamination effects.

The three principal methods available for extraction of blood samples—namely, liquid-liquid extraction, solid-liquid extraction, and column switching—are described in Chapter 3. Next chapter on selective detection methods provides an essential and overdue review of radiocolumn liquid chromatography, a technique that has shown considerable expansion during the last decade. After a brief but adequate introduction to the use of radioisotopes in chemical analysis, the chapter focuses on the use of homogenous (liquid) and heterogeneous scintillation counting and discusses the advantages of both off-line and on-line (flow through) counting.

Derivatisation is frequently used to enhance sensitivity and selectivity of detection in HPLC. Chapter 5, discusses both traditional postcolumn reactors, namely open-tubular or packed bed, and new developments in this field.

Chapter 6 examines luminescence techniques alternative to fluorescence, such as chemiluminescence (CL) and phosphorescence, for detection of nonfluorescent compounds. The use of solid-state reactor systems, including immobilised fluorophores and enzymes, has overcome many detection problems.

The volume concludes with a concise review of continuous separation techniques in flow injection analysis (FIA). The advantages of directly coupling FIA and HPLC systems include improved sensitivity (by preconcentration), selectivity (by sample cleanup or simultaneous analysis of several analytes), and increased sample throughput.

This book contains contributions from authors with internationally recognized expertise, and several chapters must be regarded as state-of-the-art-reviews. It is, therefore, an excellent addition to part A of this volume. Despite a slight overlap in concepts between the two parts the purchase of this book by chemistry libraries and laboratories using HPLC is highly recommended.

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Multidimensional Chromatography Techniques and Applications. Edited by H. J. Cortés, (The Dow Chemical Co. Midland, MI, USA), 378 pages. Marcel Dekker, New York (1990). ISBN 0-8247-8136-8. US\$ 119.50.

This book deals with hyphenated chromatography techniques and with a lot of instrumentation details for the determination of analytes of interest in complex sample matrices. Each chapter is written by experts who have practical experience in the interfacing requirements, operational details and specific application to real world samples of extreme complexity.

Chapter 1 is an excellent theoretical discussion of the limitations of one dimensional separation techniques in terms of peak capacity and peak overlap. Chapter 2 is a comprehensive, up-to-date, and thorough presentation of two dimensional TLC, including specific applications. This is a well established technique, commercial plates are available for such applications, and the theory and limitations have been well discussed.

Chapter 3 covers a rather broad topic, multidimensional gas chromatography with heavy emphasis on the instrumentation requirements. Various ways of coupling GC columns are discussed. Chapter 4 deals with selectivity tuning in capillary GC, using a battery of techniques, including mixed phases and coupled columns. There is some basic chromatographic theory about resolution and its relationship with efficiency. Specific examples of selectivity tuning are also given. Chapter 5 covers process multidimensional GC, and the specific differences which must be taken into account when using the technique for such applications.

Chapter 6 deals with multidimensional HPLC. It describes in detail how two-dimensional HPLC approaches can be automatically interfaced, how peaks can be valve switched and how fractions can be heart-cut. The chapter is very easy to follow and appreciate. Chapter 7, deals with the coupling of HPLC to capillary GC. It is very thorough, discussing the various ways of interfacing the two techniques, clearly showing the advantages and disadvantages of each method. Instrumentation is again emphasized, with diagrams and tables for a better understanding of the text.

The chapter on multidimensional SFC, written by Milton Lees' group, is a highly professional review of the entire field: what can be done with the techniques today, what has been done in the recent past, the literature available, and which specific instrumental requirements are needed for an overall success. The nature of the mobile phase requirements, pressure changes, temperature possibilities, valve switching, and other hardware areas are all very well discussed. The chapter on supercritical fluid extraction-supercritical fluid chromatography (SFE-SFC) deals with automated SFE sample cleanup and preparation prior to SFC separations. Again, instrumentation, hardware, techniques and specific applications are clearly summarized, with numerous figures and schematic diagrams.

Finally, the last chapter dealing with hardware considerations for the automation of multidimensional chromatography is very heavy on instrumentation and hardware, interfacing requirements, operational details of interfaced systems, maintenance and repair, and day-to-day details of operating multidimensional systems on a routine basis.

This treatise on multidimensional chromatography is well balanced between instrumentation and applications and should prove very useful to experienced chromatographers and those interested in expanding the possibilities of this field.

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Gas Chromatography. A Practical Course, by G Schomburg. Max-Plank-Institut für Kohlenforschung, Mulheim-Ruhr (W. Germany) 320 pages. (1990). ISBN 3-527-27879-6 (VCH, Weinheim, G.) 76 DM, ISBN 0-89573-889-9 (VCH, New York, USA).

This book is a fast and effective introduction to gas chromatography (GC), covering the main subjects and topics of modern GC. The book presentation is personal, clear and concise, even containing the depiction of tricks and traps of real world GC. However, the number of references is perhaps too reduced and they are not updated.

Following a short introduction to the basic theory of chromatographic separations, a detailed treatment of parameters and equations governing the chromatographic separations is described. Injection techniques and detection systems, as well as their potential, are overviewed. Quantitative and qualitative methods, taking into account the sources of analytical errors, are described. Elegant approaches to cope with complex mixtures, including multidimensional separations, coupling chemical reactors (i.e. for post-column derivatization and on-line hydrogenation) are nicely presented. Finally, a large variety of applications for the GC determination of different classes of compounds are described in detail, providing the gas chromatograms and analytical conditions as well.

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