Publications

Book reviews

Chemical Derivatization in Analytical Chemistry Vol. 1: Chromatography, edited by R.W. Frei and J.F. Lawrence (Plenum Press, 223 Spring Street, New York, NY, 10013, 1981, 344 pp., $39.50).

In the last few years two books, *Handbook of Derivatives for Chromatography* by K. Blau and G.S. King (Heyden and Sons Inc., 1977) and *Handbook of Analytical Derivatization Reactions* by D.R. Knapp (Wiley-Interscience, 1979) on this subject have been reviewed in this column. In contrast to the encyclopedic coverage of these texts the present book focuses on only four topics: chemical derivatization in pesticide analysis, cyclic boronates as derivatives for GC-MS or bifunctional organic compounds, general aspects of precolumn derivatization with emphasis on pharmaceutical analysis, and reaction detectors in liquid chromatography. Lawrence and Frei are also authors of an earlier volume, *Chemical Derivatization in Liquid Chromatography* Elsevier, Amsterdam, 1976, in the same general area.

Cochrane considers the gas chromatography of the pesticides under seven headings: organochlorine insecticides, herbicides, phenols, and fungicides. For the organochlorine compounds derivative formation tends toward rather drastic reaction conditions used largely for confirmatory purposes. Similarly some of the clean-up techniques tend to be rather destructive. Four primary options are offered for the organophosphorus compounds; alkaline hydrolysis followed by derivatization of the P moiety, or of the alkyl or aryl moiety, on-column transesterification, and derivatization of the intact pesticide. Several selective detectors, FPD or AFID, are of particular importance with this group of compounds. With the carbamates emphasis is on increasing thermal stability and/or increasing detectability. Once again there are four options presented. These include derivatization of either half of the molecule, on-column transesterification and derivatization of the intact molecule. In general it should be noted that on-column reactions such as transesterification or acylation tend to be rather hard on the usual GC column. The perfluorinated acyl reagents, trifluoroacetic, pentafluoropropionic and heptafluorobutyric anhydrides or imidazoles have become quite popular. Herbicides are considered under the headings: triazines, ureas, chlorophenoxy acids and miscellaneous. Cochrane manages to include 441 references in 85 pages of text to provide rather encyclopedic coverage.

The cyclic boronates of difunctional molecules are useful in terms of increased volatility and also frequently in terms of increased detector response. These derivatives are also important for GC-MS. In some cases two derivatives may be formed.

Sternson considers derivative formation for enhanced detector response, usually UV, in HPLC. The chapter is organized on a functional group basis proceeding through amines, hydroxy and phenolic compounds, carbonyl compounds, carboxylic acids, thiols and miscellaneous nitrogen containing compounds. A final section considers detector amplification by inclusion of an ion pairing reagent.

Almost one-third of the book is devoted to the final chapter by Frei on reaction detectors for HPLC. The emphasis in this case is on post column reactions. After the usual theoretical considerations of flow systems, both non-segmented and segmented streams are illustrated. An interesting balance occurs. It is not necessary that the reaction go to completion or be well defined as long as the system is highly reproducible. On the other hand the composition of chromatographic eluent seldom generates optimum reaction conditions. Consideration is given to visible, UV, fluorescence, electrochemical and radiochemical detectors. Frei's well known work with photochemical reactors is also included.

This volume is consistent with modern analytical trends. Two chapters are devoted to examples of established GC methodology and two chapters are devoted to evolving HPLC techniques. The prior handbooks by Blau and King and by Knapp provided much more systematically organized information and specific detail by emphasis was largely GC oriented. The present volume covers two narrow areas of GC analysis and two extremely broad areas of HPLC. If Sternson had provided general coverage rather than restricting emphasis to pharmaceuticals this would have been a more broadly useful book. Since this seems to be the first volume of a continuing series it would be interesting to know where the editors intend to go from here.

The results thus far are definitely uneven. This is, however, an excellent book for anyone in the HPLC area and the chapters on pre- and post-column derivatization are among the best available.

Biological/Biomedical Applications of Liquid Chromatography III, Chromatographic Science Series Volume 18, edited by G.L. Hawk, P.B. Chaplin, R.F. Hutton and C. Miol (Marcel Dekker Inc., 270 Madison Avenue, New York, NY, 1981, 420 pp., $49.75).

This appears to be a series within a series, with volumes I, II, and III of this title comprising volumes 10, 12, and 18 of the Chromatographic Science Series. Twenty-two selected papers are included from the third International Symposium of Biological/Biomedical Applications of Liquid Chromatography, Boston, MA, October, 1979. Chapters of interest to oil chemists cover prostaglandins, selected lipids and the oligosaccharides of complex glycolipids. Two factors must be taken into consideration when reading this book. To a very significant extent HPLC became a practical laboratory technique through the almost single handed long term continuous development efforts of one manufacturer. This effort included development of both equipment and first generation column materials. A very significant portion of initial published manuscripts in this field was therefore, of necessity, based on use of these materials. Making due allowance for introductory chapters, chapters not directly involving columns

or using unique homemade materials this is indeed the case in 85% of the selected manuscripts in the volumes by this title. By about 1979 second generation high efficiency column packings based on 5 micron spherical silica were widely available from a number of new suppliers. Improvements in bonding chemistry and column filling technology developed by these manufacturers increased column efficiency (theoretical plates/meter) approximately 10 fold. Through either a misfortune in timing or a pervasive parochial selection process much of the contents of this book are presently largely of historical interest. This is particularly evident in the fairly large number of chapters dealing with peptides and proteins. The 132 papers recently presented (November 1981) at the International Symposium on HPLC of Proteins and Peptides (Washington, DC) clearly indicate a mass movement away from first generation materials.

In a fast moving field rapid obsolescence is probably unavoidable. In the book under review this has to be considered if a constructive recommendation is to be made.

Organic Trace Analysis by Liquid Chromatography, by J.F. Lawrence (Academic Press, 111 Fifth Ave., New York, NY 10003, 1981, 288 pp., $34).

This is one of those books focusing on a specific area of HPLC that starts at square one and develops to whole technique before aiming at the specific application. First, the merits of LC over GC are discussed. This is followed by the usual material on theory, instrumentation, injectors, detectors, and column packings. These topics take up one-half, 142 pages, of the book. To some extent the book may be said to start at Chapter 7 with a 55-page discussion of chemical derivatization. Alternatively it may be considered to start at page 198 with aspects of sample extraction and clean-up which are particularly applicable to organic trace analysis. Integration of techniques and applications finally appear in the last two chapters. On balance, better descriptions of the general aspects of HPLC may be found elsewhere, the chapter on derivatization is reasonable, but the title of the book really only applies to the last 87 pages. Applications covered include clinical, environmental and food samples. Of possible interest to oil chemists are fatty acids in river water, aflatoxin M_1 in milk, vitamin A in dairy products, vitamin D_3 in animal feeds and vitamin E in foods and tissues.

Immobilized Enzymes in Analytical and Clinical Chemistry, Fundamentals and Applications, Chemical Analysis Series, Vol. 56, by P.W. Carr and L.D. Bowers (John Wiley & Sons Inc., New York, NY, 1981, 460 pp., $47.50).

According to the preface this volume was written as a single source for the analytical chemist. The first 147 pages therefore start at amino acid structures and continue through enzymology and enzymological techniques. Immobilization may take place by physical methods: adsorption onto water-insoluble matrix, entrapment inside water-insoluble polymer lattice, or entrapment within semipermeable microcapsule; chemical methods: attachment to derivatized water-insoluble matrix; or intermolecular crosslinking of enzyme molecules; or so called hybrid methods. Supports differ in capacity, flow characteristics, stability, ease of activation and cost with no one material outstanding in all characteristics. Various studies have found 1,000 to 10,000 fold increases in thermal stability of certain immobilized enzymes. The section on enzyme electrodes includes an interesting 17-page table of applications. Similarly the section on flow systems is entirely theoretical until one reaches a 13-page table of applications. The authors are academic (Minnesota) analytical chemists and the book appears suitable for a graduate level course. Potential readers should note that the applications referred to in the book title receive only token attention.

Lloyd A. Witting
Supelco, Inc.
Bellefonte, PA

The Infrared Spectra of Complex Molecules, Vol. 2, 2nd Edn., L.J. Bellamy (Chapman & Hall, Methuen Inc., 777 Third Ave., New York, NY 10017, 1980, 320 pp., $35).

This new book by Bellamy has been designated Volume 2, with the first book with the same title published in 1975. The new book has the sub-title of *Advances in Infrared Group Frequencies* and represents the inclusion of new material since the first book. The book needs no introduction to those chemists who employ infrared spectroscopy in their work. It is probably already on their shelves or in the libraries. I would encourage the addition of the second volume to the shelf also. It is of great value to those involved in interpretation of the infrared spectra of organic molecules.

E.G. Perkins
Department of Food Science
University of Illinois
Urbana, IL 61801

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Organophosphorus Chemistry, The Royal Society of Chemistry, Burlington House, London W1V 0BN, England; 1981; 276 pp.; $120.