

pected. With a flexible route to these new structural types available, we are examining the many possible and varied uses these molecules might exhibit.^{5,6} In a general sense, the iterative

(5) α -Linked polymeric pyrroles have useful semiconducting properties: Bryce, M. R. *Nature* **1988**, 335, 12. Cowan, D. O.; Wlygul, F. M. The Organic Solid State. *Chem. Eng. News* **1986**, 64, 28. Munn, R. W. Molecular Electronics. *Chem. Br.* **1984**, 518. Bryce, M. R. Organic Conductors. *Chem. Br.* **1988**, 781.

(6) For details of the single-crystal X-ray crystallographic structure determination of **10**, please write to Dr. J. C. Huffman, Molecular Structure Center, Indiana University, Bloomington, IN 47405. Request report no. 88158.

process depicted herein should enable an array of molecules with predictable secondary structure to become available.

Acknowledgment. The National Science Foundation and the Robert A. Welch Foundation are thanked for their financial support of this research. Dr. Douglas A. Gage, Michigan State University—NIH Mass Spectrometry Facility, is thanked for mass spectral data. Dr. Jan Wasley, Ciba-Geigy Corporation, is thanked for discussions concerning pyrrole chemistry.

Supplementary Material Available: Spectral data for compounds **1–3**, **6**, **7**, **10**, **14**, **15**, and **17** (2 pages). Ordering information is given on any current masthead page.

Additions and Corrections

Carbonylation Chemistry of the Tantalum Silyl (η^5 -C₅Me₅)-Cl₃TaSiMe₃, Synthesis, Characterization, and Reaction Chemistry of (η^5 -C₅Me₅)Cl₃Ta(η^2 -COSiMe₃) and Derivatives [*J. Am. Chem. Soc.* **1989**, 111, 149–164]. JOHN ARNOLD, T. DON TILLEY,* ARNOLD L. RHEINGOLD,* STEVEN J. GEIB, and ATTA M. ARIF

The crystal structure of Cp*Cl₃Ta[η^2 -OC(PEt₃)SiMe₃] (**6**) was originally reported as a fully mirror-plane disordered structure in the orthorhombic space group *Pcam* (*R* = 6.77%). A reexamination of the structure reveals that the correct space group is *Pca*2₁ (*R* = 2.85%) and that an ordered and chemically more reasonable structure is obtained, although all significant features of the earlier report remain unchanged. A set of the redetermined data may be obtained from one of us (A.L.R.) or from the complete disclosure that has been submitted for publication (Rheingold, A. L. *Acta Crystallogr.*, Sect. C).

Book Reviews*

Flow Injection Atomic Spectroscopy. Practical Spectroscopy Series. Volume 7. Edited by José Luis Burguera (University of Los Andes, Venezuela). Marcel Dekker: New York and Basel, 1989. xii + 353 pp. \$125.00. ISBN 0-8247-8059-0.

More than 10 years has passed since the publication of the first papers on flow injection analysis (FIA), and the technique has now been clearly shown to have many widespread applications in analytical chemistry. One of these important applications, of course, is atomic spectroscopy, and therefore, this book comes along at a very appropriate time. This book provides a wealth of information for a basic understanding of the flow injection analysis-atomic spectroscopy (FIA-AS) technique. The book consists of a Foreword written by the originator of the FIA technique, Professor Jaromir Růžička, a Preface written by the Editor, eight chapters written by a variety of authors, and two very useful appendices. There is also an author and a subject index.

The first chapter, written by Kent K. Stewart, gives a general introduction to the technique and describes some basic components of FIA-AS systems. Various types of FIA-AS assay systems are described. In the second chapter, William E. van der Linden discusses some theoretical aspects of the technique, including some specific aspects related to the use of a flame atomic-absorption spectrophotometer as a detector. In Chapter 3, Jacobus F. van Staden describes basic components including sampling, pumping, manifold, and nebulizer-burner systems. Chapter 4, by Khaolun Fang, discusses various analytical methods and techniques, including hydride generation methods and cold vapor methods for mercury; and Chapter 5, by Miguel Valcárcel and Mercedes Gallego, describes separation techniques including continuous precipitation, liquid extraction and ion exchange. Chapter 6, by Elias A. G. Zagatto and co-workers, describes some selected applications of FIA-AS in agricultural and environmental analysis; and Chapter 7, by Roy A. Sherwood and Bernard F. Rocks, describes applications of the technique in clinical

chemistry. Some specialized applications involving graphite furnaces, chromatography, and inductively coupled plasma (ICP) atomic emission spectrometry are discussed. The final chapter, by Marcela Burguera, José Luis Burguera, and Gilbert E. Pacey, provides some useful information as to "current trends" in FIA-AS including instrumental developments such as speciation, conversion, automation, and miniaturized FIA systems. Some recent applications are also considered. There is also a somewhat subjective discussion of the present and the future of the technique. The book concludes with two useful appendices, Appendix A is a list of symbols, and Appendix B is an FIA-AS bibliography. Overall, this book is highly recommended.

Peter N. Keliher, Villanova University

Introduction to Microscale High-Performance Liquid Chromatography. Edited by Daido Ishii (Nagoya University). VCH: New York and Weinheim, 1988. xii + 208 pp. \$59.95. ISBN 0-89573-309-9.

This book consists of 7 chapters written by different contributors and 10 appendices which list the available packing materials for the preparation of packed and microcolumns. The editor, D. Ishii, is the co-author on four of the seven chapters. Chapter 1 gives a brief introduction on microcolumn HPLC (~5 pages), Chapter 2 describes the instrumental requirement in microcolumn HPLC (24 pages), Chapter 3 covers the characteristics of microcolumns (34 pages), Chapter 4 describes the use of different common LC detectors (UV, fluorescence, and electrochemical) in microcolumn HPLC experiment (23 pages), while chapter 5 (17 pages) covers the hyphenated systems (microcolumns with IR and MS detection). Chapter 6 is focused on the use of postcolumn derivatization in microcolumn HPLC mostly on the band broadening due to the use of different postcolumn reactors. Finally in Chapter 7 different applications of microcolumn HPLC is described rather extensively as compared to the first six chapters (52 pages).

All chapters are well written, and there seems to be no typographical errors.

*Unsigned book reviews are by the Book Review Editor.

The references are surprisingly outdated. With the exception of a couple of Buyers Laboratory Guides from 1986 and a few papers from 1985, most of the literature cited belongs to 1984 and the prior years. Note that in 1984 and 1985 two books on the same topic were published, one of which was coedited by the same editor.

Morteza G. Khaledi, North Carolina State University

Tables of Spectral Data for Structure Determination of Organic Compounds. Second Edition. By Ernő Pretsch (Eidgenössische Technische Hochschule) et al. Springer-Verlag: New York and Berlin. 1989. various pagings. \$39.50. ISBN 0-387-51202-0.

This remarkably useful book is a compilation of spectroscopic data in compact yet easily used form. It begins with 75 pages of summary tables of the essential ^{13}C and ^1H NMR, IR, and UV/vis characteristics of a substantial range of structural features. Then comes a section of "combination tables", in which the characteristics of types of functional groups are set out; here one can find, for example, generalized data on NMR, IR, UV/vis, and MS of nitro compounds, ethers, etc., with cross-references to further details. Finally, each form of spectroscopy is allotted a "chapter" to tables in which the spectra of a satisfyingly large number of specific examples are set out. For example, under ^{13}C NMR, Nitriles, there are given the shift values for each carbon of acetonitrile, propanonitrile, isobutyronitrile, pivalonitrile, acrylonitrile, malononitrile, succinonitrile, cyclohexanecarbonitrile, and benzonitrile. The UV/vis spectra are graphic.

The arrangement is intrinsically logical, and the table of contents is sufficiently detailed, such that desired information is easy to find. However, there is also a subject index.

This is a very handy compilation that most organic chemists will delight in owning.

Reviews of Environmental Contamination and Toxicology. Volumes 100-104. Edited by George W. Ware. Springer-Verlag: New York and Berlin. 1987. Volume 100: xi + 156 pp. \$41.00. ISBN 0-387-96583-1. Volume 101: viii + 169 pp. \$43.00. ISBN 0-387-96593-9. 1988. Volume 102: x + 160 pp. \$38.00. ISBN 0-387-96647-1. Volume 103: ix + 158 pp. \$38.00. ISBN 0-387-96693-5. Volume 104: viii + 225 pp. \$38.00. ISBN 0-387-18629-8.

Volume 100 of this series of reviews contains a cumulative table of subjects covered in Volumes 1-100, a subject index in detail for Volumes 91-99, and a cumulative index of contributing authors for Volumes 1-100. It should enhance the reference value of the series.

Volume 101 contains five reviews, four of which deal with chlorinated organics: 2,4-dichlorophenoxyacetic acid, trichloroethylene, hexachloro-1,3-butadiene, and polychlorinated biphenyls. The other review is on cleaning protective apparel.

Volume 102 contains three reviews on somewhat unrelated topics: teratologic testing, cellular toxicology, and bioaccumulation of persistent organic chemicals in aquatic organisms.

The three reviews in Volume 103 are about Henry's Law constants for pesticides, photochemistry of herbicides in the environment, and partition of organic compounds in aquatic systems.

The content of Volume 104 consists of the texts of Health Advisories issued by The Office of Drinking Water of the U.S. Environmental Protection Agency for 16 pesticides. They are accompanied by a list of acronyms and abbreviations and an introduction. The pesticides range from Alachlor to Toxaphene, and include Aldicarb, Chlordane, ethylene bromide, Lindane, and pentachlorophenol.

Each of Volumes 101-104 is provided with a subject index.

Theilheimer's Synthetic Methods of Organic Chemistry. Volume 32. Edited by A. F. Finch. S. Karger: Basel. 1989. xxii + 506 pp. \$465.00. ISBN 3-8055-4896-6.

This yearbook 1989 covers papers published in 1987 and early 1988. The pattern and detail of coverage keeps to the successful precedent of the earlier volumes of the series: brief experimental descriptions and equations with structural formulas, all arranged in a highly systematic way.

One of the features of every volume is a short essay, Trends in Synthetic Organic Chemistry. Although it is only 5 pages long in this volume, it is packed with information, and it serves to bridge the gap between the formal, systematic coverage and the date of printing of the book. Thus, it is devoted largely to the literature of 1989 and late 1988. Another consistent feature of the series is the index, which is extraordinarily thorough and provides an alternative means of access for those who do not want to learn the systematic organization of the data in this book.

As has been noted in reviews of previous volumes, this series is expensive. However, one is paying for a service, an enormous amount of work in searching, compiling, and organizing. It should save much more in the valuable time of a research chemist than the cost of the volumes to the institutional or company library. And although it is essentially a reference work, a key to the literature, it is also interesting and instructive simply to browse in it.

X-ray Structure Determination. A Practical Guide. Second Edition. By G. H. Stout and L. H. Jensen (University of Washington). John Wiley and Sons: New York. 1989. xv + 453. \$45.00. ISBN 0-471-60711-8.

The first edition of this text appeared in 1968. In the intervening period several other texts have appeared that also emphasize practical aspects, to the extent of leading the reader through the process of preliminary studies, data collection, and structure solution for specific compounds. This much-awaited second edition preserves the clarity of exposition and the generality and practicality that were the hallmarks of the first edition. Numerous numerical examples and figures, the vast majority are retained from the first edition, illuminate the text. The book is aimed at the nonspecialist X-ray crystallographer. A cautious approach involving preliminary characterization by precession or Weissenberg photography is still advocated. However, for the increasing number of laboratories that lack or do not use such equipment the goal of sound diffractometry may be less than fully met: while many informative anecdotes are provided, there remains a lack of general principles and specific procedures that need to be applied to prevent becoming another anecdote or being exhibited in the Marsh-Shoemaker-Herbststein rogues' gallery of defective crystal structures.

Part I (Preliminary Stages) describes sources and monochromatization of X-rays (the price given for X-ray tubes is \$1000 more than this reviewer pays), the diffraction of X-rays by crystals, crystal symmetry and space groups, crystal properties, space group and unit cell determination by various film methods, intensity measurements (at least some modern counters are linear to better than three times the 50000 counts/s rate given in both first and second editions), data reduction, and the Fourier relationships between the structure factors and electron density. Part II (The Phase Problem) describes in excellent detail solutions to the phase problem by direct methods, Patterson methods, heavy atom isomorphous replacement and anomalous dispersion methods, and search methods; the elaboration of the structure completes Part II. Part III (Refinement of Crystal Structures) covers refinement by Fourier syntheses, location of hydrogen atoms, refinement by least-squares techniques, random and systematic errors, and analysis of derived results. Several appendices follow covering Bragg's law, the general (ellipsoidal) temperature factor expression, matrix operations, structure factor graphs and Fourier transforms (a curious anachronism from precomputer days resurrected for the second edition), and weighting functions.

Relative to the first edition, there are sections added on synchrotron radiation sources, area detectors, diffractometer theory and practice, and the fast Fourier transform; the chapters on space group symmetry and direct methods have been strengthened and lengthened; and a substantial body of material (~40 pages) on film methods eliminated. The structure of the book and much of the text are unchanged from the first. Many useful comments are interjected as footnotes; references have been thoroughly revised and updated to 1988; a large number of key post-1968 references have been added, while references to the primary literature for seminal papers have been retained. The net result is, remarkably, a somewhat shorter text than previously.

With the demise of Weissenberg methods and the resurgence of very small rotation photography for protein work, it is unfortunate that the still considerable space allocated to Weissenberg techniques was not allocated in one part to the indexing of rotation photographs and in the other part to the de Jong-Bowman technique that complements the standard geometry for precession photography. With the increasing reliance of many practitioners on the diffractometer and its software for the identification and even determination of space groups, it is again unfortunate that more attention was not given to the mechanics of cell reduction and the detection of higher symmetry lattices, along with examples—essentially only references are given. This is particularly pertinent since this reviewer has many examples of the failure of the cell reduction package offered in one widely used, commercially available suite of crystallographic programs. Another shortfall is the inadequate description of the various methods and pitfalls associated therewith for absorption corrections. While many tricks of the trade are given on other subjects, there are relatively few given on how to force direct methods to work on a recalcitrant set of data. Solutions to the phase problem are well-covered but corresponding details on phase extension and refinement procedures are absent.

As a teaching text, this book lacks exercises at the end of each chapter, leaving the instructor to provide his own, or to raid from another source.

This and some of the other criticisms given above are probably more a matter of this reviewer's personal preferences than a fundamental fault.

The perfect text, of course, remains to be written. However, this second edition is a very good one, retaining the strengths and clarifying the (few) infelicities of the first and incorporating some of the advances made in the interim along with numerous salutary lessons on the dangers of handing over one's critical faculties to a user-friendly computer. Will the percentage of defective crystal structures appearing in the literature decrease as a result of this book? Alas, probably not.

Geoffrey B. Jameson, *Georgetown University*

Iron Porphyrins Part 3. Physical Bioinorganic Chemistry 4. Edited by A. B. P. Lever (York University) and H. B. Gray (California Institute of Technology). VCH: New York. 1989. xiii + 309 pp. \$59.50. ISBN 0895-73718-3.

Iron Porphyrins Part 3 completes that part of the VCH Publishers series on Physical Bioinorganic Chemistry which is devoted to the physical techniques used in characterizing iron porphyrins and heme proteins. The editors have directed contributors to emphasize established theory and "avoid inclusion of speculative materials so that this series will remain current and valid for many years". *Iron Porphyrins Part 1* and *Part 2*, which were published in 1983, have admirably fulfilled this goal and remain excellent reference works with exceptional pedagogical value to the researcher using allied techniques. *Part 3* now extends this repertory to include magnetic circular dichroism spectroscopy, Mössbauer spectroscopy, and X-ray absorption spectroscopy.

Dawson and Dooley introduce their chapter on magnetic circular dichroism spectroscopy with enough theory to show the experimentalist what information MCD can deliver and how it is obtained. MCD offers advantages over magnetic susceptibility measurements as a probe of paramagnetism, especially for multiheme proteins, and it complements EPR spectroscopy making it easier to study even-spin systems. A review of hemoglobin and myoglobin studies is followed by coverage of dioxygenases, cytochromes *b*, *c*, and *f*, peroxidase, catalase, chloroperoxidase, and other simple (noninteracting) heme proteins. MCD probing of the axial ligation of cytochrome P-450 and cytochrome oxidase as a multiheme example receive special attention. An addendum to this chapter apparently became necessary due to processing delays and is used to update the literature reviewed. The 102 references and the several new heme proteins covered in the addendum attest to the increasing value of MCD spectroscopy.

Mössbauer spectroscopy is a powerful diagnostic tool for determining the oxidation number and spin states for iron in heme proteins. In principle it can also be used to deduce model wave functions to provide insight into electronic structure of iron porphyrin complexes. Debrunner concedes in his review that this second, more fundamental analysis is exceedingly difficult. The formalism required to analyze and interpret a Mössbauer spectrum is succinctly summarized, and an abbreviated section on experimental considerations is presented. Mössbauer experimental techniques for biochemical studies are not discussed. Debrunner organizes most of the chapter under spin case and iron oxidation state headings, beginning with ferrous low spin and progressing through ferryl porphyrins and other higher oxidation states. Although the ferric intermediate spin, $S = 3/2$, and the ferrous intermediate spin, $S = 1$, porphyrins are relatively rare, they are discussed since their existence and their properties have important implications for other iron porphyrins. Coverage of iron porphyrin model compounds, porphyrin dimers, and spin-coupled systems counterbalances the emphasis on heme proteins. Debrunner has also updated his review by appending a section on Iron-Porphyrin Literature to Mid 1987.

Penner-Hahn and Hodgson give a concise description of the physical principles of X-ray absorption spectroscopy and discuss practical details for collecting and interpreting EXAFS and X-ray absorption edge data. This overview clarifies some of the experimental constraints as well as champions the technique's unique power for providing direct structural information about the absorbing site. A review of the EXAFS and XANES literature on model porphyrin compounds leads into the discussion of the important contributions of X-ray absorption spectroscopy to studies of hemoglobin. Electron transfer hemoproteins, cytochrome P-450, chloroperoxidase, horseradish peroxidase, and cytochrome *c* oxidase are discussed. The authors point out that the method is ripe for both experimental and theoretical advances and is likely to become more accessible with the development of new synchrotron facilities.

Iron Porphyrins Part 3 is a worthy addition to this survey of physical techniques applicable to the study of iron porphyrins and will be valued by researchers and students in this or related fields.

Richard L. Petersen, *Memphis State University*

Advances in Electrophoresis. Volume 1. Edited by A. Chrambach (National Institutes of Health) et al. VCH: Weinheim and New York. 1988. ix + 441 pp. \$116.50. ISBN 0-89573-669-1.

The editors of this volume state that the purpose of *Advances in Electrophoresis* is to serve as a "central review bank" to assemble the multitude of reviews and proceedings appearing each year on various aspects of electrophoresis. If subsequent volumes meet this goal as well as Volume 1 has, this series will serve as a valuable reference in the field. The editors have assembled an excellent list of authors to survey particular areas of interest. They have also struck a fine balance between technique-oriented and application-oriented reviews. Volume 1 provides a good overview of the state of the art for electrophoresis.

The first and longest chapter provides an excellent discussion of 2-D PAGE. This is appropriate as this is the most popular electrophoretic technique. Both theory and practice are described in a manner useful to both the novice and experienced user of electrophoresis. Aspects of detection and data analysis are presented as well as the typical discussion of separation. This volume is a worthy acquisition for anyone using electrophoresis for this chapter alone.

Much attention is then provided to protein and nucleic acids in Chapters 2-5. These chapters cover such topics as detection of proteins, blotting techniques, separation of DNA, and affinity techniques. The chapter on protein detection is most welcome as it describes quantitation as well as mere detection. This topic is often overlooked in other sources.

Chapter 6 covers the topic of isotachoporesis in detail. The authors properly discuss both the problems and the potential of this method. The chapter contains a rather detailed description of the theory, which may put off the casual user, but also provides nice descriptions of several commercial instruments.

Chapter 7 describes techniques for preparative PAGE. This is a nice presentation but could have been better placed earlier in the volume. The final chapter provides an example application of electrophoresis in forensic science. Surprisingly, there is no discussion of the technique of capillary zone electrophoresis which has shown such promise recently. This may be because of the lag time between writing and publication and will hopefully be corrected with Volume 2.

Overall this volume will be most useful to workers relatively new to the area. Experienced users may find much of the presentation well known, as is the nature of reviews, but there is still much insight to be gleaned from these presentations. This volume deserves a spot in any collection on electrophoresis.

Craig E. Lunte, *University of Kansas*

Chemical Reactions in Plastics Processing. By Klaus Kircher (Translation by Carla B. Grot). Oxford University: Oxford and New York. 1987. x + 214 pp. \$45.00. ISBN 0-02-947520-1.

The molding of plastic articles involves, primarily, the physical process of heating and cooling a linear high molecular weight polymer. However, about one-fourth of all processing methods employ chemical reactions. The final products are manufactured from monomers and low molecular weight prepolymers. This book provides a detailed survey of the chemical reactions which occur during polymer processing.

After a brief introduction in the first five chapters, these reactions are grouped according to the methods of synthesis. The mechanism and kinetics of each polymerization reaction are discussed at the beginning of each chapter.

Chapter 6 covers free-radical polymerizations. Processing reactions include PMMA, unsaturated polyesters, diallyl esters, plastics of high distortion temperature such as polyimide, and polyamides from ϵ -caprolactam.

Chapter 7 deals with polycondensation reactions, which include phenolic and amino resins. The formation of polycarbodiimide is a polycondensation reaction. Since it is the reaction product of isocyanate, the author put this reaction in Chapter 8, which discusses polyaddition reactions. The reactions in polyurethanes, polyisocyanurates, and epoxy polymers are presented in detail.

Chapters 9-11 cover the cross-linking of polyethylene, polypropylene, fluoro polymers, diene polymers, and others.

The last chapter is devoted to the Degradation of Polymers during Processing. The discussion emphasizes the mechanism of degradation.

This is a very useful book; the structure and formula of chemicals are well illustrated. The book is highly recommended for chemical engineers working in the polymer processing industry, and it serves as an introduction to polymer chemistry for polymer students. The book is written in such a way that a nonchemist can also understand it.

Shaio-wen Wong, *University of Detroit*