The mother liquor from the above crystallization was evaporated in vacuo, the residue was take up in ethyl acetate (200 mL), and the solution was shaken with 2 N sulfuric acid (75 mL). The organic phase was combined with an ethyl acetate extract of the aqueous phase, and the solution was washed with water and dried (magnesium sulfate). The solvent was removed in vacuo and the partially resolved acid (8 g, 31.4 mmol) was dissolved in ethanol (50 mL) and added to a suspension of cinchonine (9.24 g, 31.4 mmol) in hot ethanol (200 mL). The resulting solution was heated at reflux temperature for 0.5 h, the solvent was removed in vacuo, and the residual oil was dissolved in ethyl acetate (150 mL). After 2 h, the salt that crystallized [3.4 g; mp 174–175 °C; $[\alpha]_D$ +267° (c 1, MeOH)] was collected by filtration and recrystallized from ethyl acetate (75 mL). The pure cinchonine salt (2.13 g, 25%) had the following: mp 178–180 °C; $[\alpha]_D$ +271° (c 1, MeOH); UV 227, 245 (sh), 313 nm (ε 40700, 8910, 21400). Anal. ($C_{34}H_{35}N_3O_4$) C, H, N.

Dilute sulfuric acid (25 mL, 2 N) was added to a suspension of the above cinchonidine salt (4.7 g) in water (50 mL). The solution was extracted with ethyl acetate, and the extract was washed with water, dried, and evaporated in vacuo. The solid residue was recrystallized from hexane–ethyl acetate to give pure (−)-ketorolac (1.74 g, 80%): mp 169–170 °C; $[\alpha]_D$ −176° (c 1, MeOH). Anal. ($C_{15}H_{13}NO_3$) C, H, N.

Decomposition of the cinchonine salt in the manner described above gave the crude (+)-acid, which on crystallization from hexane–ethyl acetate gave pure (+)-ketorolac (75% yield): mp 174 °C; $[\alpha]_D$ +173° (c 1, MeOH). Anal. ($C_{15}H_{13}NO_3$) C, H, N.

To determine the effectiveness of the resolution of ketorolac, each antipode was esterified with excess ethereal diazomethane, and the esters were subjected to analysis by NMR spectroscopy in the presence of the chiral shift reagent $Pr(hfc)_3$. The racemic ester (0.030 g/0.6 mL of $CDCl_3$) showed two singlets for the enantiotopic methyl groups at δ 2.76 and 2.90 in the presence of the shift reagent (0.062 g). The (−)-methyl ester showed only one singlet at δ 2.90, indicating 100% enantiomeric purity. The enantiomeric purity of the (+) isomer can thus be calculated to be >98% on the basis of its optical rotation.

Synthesis of the Amides 2a and 2b of (+)-(*R*)-1-(1-Naphthyl)ethylamine and (−)- and (+)-5-Benzoyl-1,2-dihydro-3*H*-pyrrolo[1,2-*a*]pyrrole-1-carboxylic Acids, Respectively. To a stirred suspension (0.51 g, 2 mmol) of (±)-1 and dicyclohexylcarbodiimide (0.452 g, 2.2 mmol) in dry dichloromethane (20 mL) was added the amine (0.32 mL; Aldrich Gold label, distilled).[15] After 4 h, this mixture was diluted with additional dichloromethane (30 mL) and filtered. The filtrate was washed successively with 1 N hydrochloric acid, 5% sodium bicarbonate solution, and water. Evaporation of the dried organic phase gave an oil, which solidified on standing. The diastereoisomeric mixture was separated by flash chromatography on silica gel (100 g) with hexane–ethyl acetate (3:2) as the eluting solvent. The less polar and more polar amides **2a** (0.282 g, 35%) and **2b** (0.251 g, 31%) were obtained as solids. In separate experiments, the amide **2a** (R_f 0.5, silica gel, 1:1 hexane–ethyl acetate) was obtained from (−)-1 and **2b** (R_f 0.3) was prepared from (+)-1.

On crystallization from ethyl acetate, **2a** had the following: mp 212–213 °C; $[\alpha]_D$ −164° (c 0.5, $CHCl_3$); UV 225, 248, 271, 281, 293, 311 nm (ε 74100, 8320, 8710, 11200, 14100, 19100); IR (KBr) 3285, 1640, 1630 cm^{-1}; NMR δ 1.60 (d, 3 H, J = 7 Hz), 2.78 (q, 2 H, J = 7.5 Hz), 3.80 (t, 1 H, J = 7.5 Hz), 4.20–4.70 (m, 2 H), 5.70–6.10 (m, 2 H), 6.10–6.40 (1 H, exchanged with D_2O), 6.76 (d, 1 H, J = 4.5 Hz), 7.30–7.70 (m, 7 H), 7.70–8.20 (m, 5 H). Anal. ($C_{27}H_{24}N_2O_2$) C, H, N.

Crystallization of **2b** from hexane–ethyl acetate gave a solid: mp 222–223 °C; $[\alpha]_D$ +126° (c 0.5, $CHCl_3$); UV 224, 248, 271, 281, 293, 312 nm (ε 69200, 8320, 8510, 11200, 13500, 18200); IR (KBr) 3325, 1670, 1595 cm^{-1}; NMR δ 1.70 (d, 3 H, J = 7 Hz), 2.80 (q, 2 H, J = 7.5 Hz), 3.86 (t, 1 H, J = 7.5 Hz), 4.15–4.65 (m, 2 H), 5.73–6.07 (m, 2 H), 6.10–6.35 (1 H, exchanged with D_2O), 6.75 (d, 1 H, J = 4.5 Hz), 7.30–7.70 (m, 7 H), 7.70–8.20 (m, 5 H). Anal. ($C_{27}H_{24}N_2O_2$) C, H, N.

Acknowledgment. One of us (R.A.T) thanks Dr. M. Soriana-Garcia for helpful discussions and for his assistance in the crystal structure determination and A. Cuellar for technical assistance.

Supplementary Material Available: Bond lengths and bond angles (Table II), final atomic coordinates and U_{eq} values (Table III), hydrogen coordinates (Table IV), and anisotropic temperature factors (Table V) (8 pages). Ordering information is given on any current masthead page.

(14) The elemental analysis of the new compounds described in the Experimental Section were within ±0.4% of the calculated values.

(15) Mori, K.; Nukada, T.; Ebata, T. *Tetrahedron* 1981, *37*, 1343.

Book Reviews

The Chemistry and Biology of Isoquinoline Alkaloids. Edited by J. D. Phillipson, M. F. Roberts, and M. H. Zenk. Springer-Verlag, Berlin-Heidelberg-New York-Tokyo. 1985. vii + 304 pp. 17 × 23 cm. ISBN 3-540-13980-X. $39.00.

The Chemistry and Biology of Isoquinoline Alkaloids is the proceedings volume edited on the basis of the plenary lectures presented at a 3-day symposium arranged by the Phytochemical Society of Europe in London in April 1984. The chapters cover the recent progress of research on the isolation, structure elucidation, synthesis, pharmacology, structure–activity investigation, and biosynthesis and catabolism, as well as production by plant cell culture techniques, of the isoquinoline alkaloids. The titles of lectures and the authors are as follows:

"Plants as a Source of Isoquinoline Alkaloids" (N. G. Bisset), "Chemotaxonomy of the Papaveraceae Alkaloids" (V. Preininger), "Structure Activities and Pharmacological Properties of the Opium Alkaloids" (E. Lindner), "The Occurrence of Simple Isoquinolines in Plants" (J. Lundstrom), "*Erythrina* Alkaloids" (A. H. Jackson), "Annonaceae Alkaloids" (A. Cave), "The Chemistry and Pharmacology of Cularine Alkaloids" (L. Castedo), "Bisbenzylisoquinoline Alkaloids" (P. L. Schiff, Jr.), "Natural Degradative Routes for the Aporphines" (M. Shamma), "Synthesis and Structure–Activity Relationships of Aprophines as Dopamine Receptor Agonists and Antagonists" (J. L. Neumeyer), "The Chemistry and Pharmacology of Morphinan Alkaloids" (A. Brossi), "The Development of a Practical Total Synthesis of Natural and Unnatural Codeine, Morphine and Thebaine" (K. C. Rice), "Biomimetic and Total Synthesis of Monoterpenoid Isoquinoline Alkaloids" (R. B. Herbert), "Biosynthesis of Morphinan Alkaloids" (E. Brochmann-Hanssen), "Enzymology of Benzylisoquinoline Alkaloid Formation" (M. H. Zenk), "Morphinan Alkaloids from Plant Cell Cultures" (F. Constabel), "The Production of Isoquinoline Alkaloid Accumulation" (T. M. Kutchan, S. Ayabe, C. J. Coscia).

The volume also includes a brief subject index.

All the authors describe their topic where they have made considerable contribution in detail and standards. In addition to the referecnes, the majority of the chapters contain suggested reading for the topic reviewed that helps the readers in broadening their knowledge on the particular group of alkaloids.

In summary, *The Chemistry and Biology of Isoquinoline Alkaloids* presents an interesting and current discussion of a

number of fields of topical interest in isoquinoline chemistry and pharmacology. It contains an outstanding treatment of the chemistry of isoquinoline alkaloids presented by active researchers in the field. Undoubtedly, it should be in every library of scientists working in the field of natural products and in general libraries of medicinal chemistry and pharmacology. Alkaloid chemists will welcome this collection of up-to-date manuscripts.

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Analysis of Neuropeptides by Liquid Chromatography and Mass Spectrometry. Dominic M. Desiderio. Vol. 6 of the Series Techniques and Instrumentation in Analytical Chemistry. Elsevier, New York. 1984. xviii + 235 pp. 17 × 25 cm. ISBN 0-444-42418-0. $63.50.

This book is intended to provide a communication link between biological and clinical investigators and analytical chemists, specifically mass spectrometrists, all of whom are engaged in some aspect of neuropeptide research. The title implies that the book is limited to LC and MS but other parallel methods are also described in some detail and evaluated. LC–MS as a combined technique is considered only in the last chapter, which contains a fairly complete survey of the various approaches under development.

The book is divided into eight chapters. (1) Introduction, (2) Neuropeptides, (3) Biochemical Sampling Techniques, (4) Reverse Phase Chromatography of Peptides, (5) Analytical Measurements of Endogeneous Peptides (RIA, Bioassay, RRA), (6) MS of Peptides, (7) Measurement of Endogeneous Biological Peptides with MS, (8) Instrumental Developments (High Mass, negative ions, LC–MS).

For those who are entering into or are engaged in these areas of research, the availability of this text means a saving of effort in the establishment of general principles and provides a starting point for literature searches of more specific areas. The involved researcher will obviously wish to pursue selected areas in more detail. The author has himself become quite familiar with the physiology, biochemistry, and analytical chemistry needed for this type of work. He gives lucid explanations that can be followed by a newcomer and a realistic assessment of the power and potential problems of the various techniques. The book thus fulfills its stated purpose of collecting together sufficient introductory material to inform the novice about the basic principles in each of the aspects of the work.

There are useful tables of definitions and abbreviations and an adequate index. These are important to the reader because terminology is sometimes employed in the text before it is defined. More careful editing would have been helpful, particularly in Chapter 5. Some discussions recur at different places in the book, e.g., HPLC of peptides in Chapters 2 and 3. Sections that consist of one sentence summaries of each of a series of papers are tedious to read. For these sections, an evaluation of the general area would have been more informative.

In addition to long discussions of his own work, including extremely detailed descriptions of data in papers not (at that time) yet accepted for publication (pp 81–88, 96–107, much of Chapter 7), the author chose to quote his own work when more appropriate references are available. In Chapter 5, an example is given of using *visual* LRMS observation of an oscilloscope MH^+ peak as verification of an RIA assay, an approach that surely must have bothered its reviewers. He also presents his faint FABMS spectrum of insulin when much better quality spectra of this compound had already appeared. He cites three of his papers as the only references on photoplate MS when extensive (and much earlier) work was published by others. Instrumentation not utilized by the author such as quadrupole (especially triple quadrupole) MS and FTMS receive only minimal attention.

This volume is devoted to areas of interest to neurobiologists and analytical chemists. The selection of the material is however too much influenced by the author's personal research interest to represent an objective survey of the field.

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Bioactivation of Foreign Compounds. Edited by M. W. Anders. Academic Press, New York. 1985. xv + 555 pp. 16 × 23.5 cm. ISBN 0-12-059480-3. $85.00.

This book is an additional volume of a series of monographs in Biochemical Pharmacology and Toxicology published by Academic Press. The earlier monographs, edited by W. B. Jakoby, presented an overall view of detoxication mechanisms including general aspects of the mixed function oxygenase systems. This present volume is more specific and contains a series of excellent reviews on the chemical mechanisms by which foreign compounds are bioactivated to form toxic metabolites.

The volume is organized into two parts. Part I contains three reviews on general aspects of bioactivation of foreign compounds. The first review appropriately written by E. C. Miller and J. A. Miller discusses succinctly early observations on the bioactivation of various foreign compounds to reactive electrophiles. These authors are to be commended for unifying much of the early literature and giving a comprehensible summary of the postulated mechanisms for the bioactivation of foreign compounds. The second chapter by J. R. Gillette describes in general terms the pharmacokinetics for the formation of reactive metabolites including a discussion on short, intermediate and long-lived chemically reactive metabolites. The addition of simulations help further to clarify the theoretical equations. The third chapter of D. J. Reed describes some of the cellular defense mechanisms against reactive metabolites. Included in this chapter are discussions of various protection mechanisms by the organism against such reactive intermediates as electrophiles and free radicals. From this chapter, the reader gains a sense of hope for the future development of agents to counteract exposure to foreign compounds which are bioactivated.

The second section of this volume contains reviews which are more specific in scope and discuss bioactivation by chemical class. Chapters in this section include the bioactivation of alkanes (J. S. Bus), alkenes and alkynes (P. R. Ortiz de Montellano), benzenes (L. S. Kaminsky), polycyclic aromatic hydrocarbons (D. R. Thakker et al.), furans (L. T. Burke and M. R. Boyd), phenols, catechols, and quinones (R. D. Irons and T. Sawahata), halogenated alkanes (M. W. Anders and L. R. Pohl), halogenated alkenes and alkynes (D. Henschler), arylamines and arylamides (S. D. Nelson), arylhydroxylamines (P. E. Hanna and R. B. Banks), nitrosamines (M. C. Archer and G. E. Labuc), hydrazines (R. A. Prough and S. J. Moloney), nitroimidazoles (P. D. Josephy and R. P. Mason), nitriles (A. E. Ahmed et al.) and thiono sulfur compounds (R. A. Neal). Each of these reviews are well written and organized, replete with examples. In addition each chapter is prefaced with a table of contents. The references appear to be complete and fairly recent articles are cited.

In summary, this volume contains a rather complete review of the subject of bioactivation of foreign compounds. The editor should be commended for obtaining authors who are not only well versed in their subject but have the ability to write clearly. The information in this volume is very comprehensive. Thus, this volume is highly recommended to those persons who are either performing research in the field or would like to gain more insight in this all important subject.

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The Alkaloids. Volume 24. Edited by Arnold Brossi. Academic Press, New York. 1985. xx + 359 pp. 16 × 23 cm. ISBN 0-12-4695-24-8.

This volume devotes over one-third of the pages to two important groups of isoquinoline alkaloids, "The Aporphine Alkaloids", reviewed in Vols. IV, IX, and XIV, and "The Phthalideisoquinoline Alkaloids", reviewed in Vols. VII and IX and also discussed in a more general way in Vol. XVII. They have now been brought up to date, and are presented in detail in this volume.

The discussion of the Aporphine Alkaloids (T. Kametani and T. Honda) could have been more current. Few references are cited beyond 1981. A brief paragraph highlights some of the phar-

macological activities of these alkaloids. The Phthalideisoquinoline Alkaloids (D. B. MacLean) are adequately reviewed and are of interest to medicinal chemists primarily due to the interest of (+)-bicuculline as an antagonist of γ-aminobutyric acid (GABA).

"*Aristotelia* Alkaloids" (W. C. Taylor) and "*Eupomatia* Alkaloids" (R. C. Bick and M. A. Hai) predominantly originating from plants of the Australian continent are appropriately reviewed by Australian chemists.

A chapter on "Marine Alkaloids" (C. Christophersen) represent a large and fast-growing group of natural products of the marine habitat, not explored in depth and possibly containing many novel substances of interesting pharmacological properties. The state of the art of this interesting group of compounds is covered up to 1984.

A concluding chapter on "The Study of Alkaloids by Spectral Methods" (R. J. Highet and J. W. Wheeler) is the second of a series of articles which appeared recently in this treatise and describes modern physical methods important for the structure determination and characterization of alkaloids.

A subject index for this volume as well as the contents of previous volumes are included in Volume XXIV.

Staff

Handbook of Tritium NMR Spectroscopy and Applications. By E. A. Evans, D. C. Warrell, J. A. Elvidge, and J. R. Jones. Wiley, New York. 1985. xi + 249 pp. 15 × 23 cm. ISBN 0471-9053-6. $39.95.

Tritium has become an exceptionally valuable and popular radiolabel for many compounds, especially in the life sciences. Although the first tritium NMR spectrum was obtained as early as 1947, it has only been in the last 10–15 years with improved instrumentation that this technique has been refined to become a practical and informative tool for the analysis of tritium-containing compounds. A number of excellent reviews have been written by these authors on tritium NMR, but to my knowledge this is the first book to appear on the subject.

Divided into three chapters, this volume first discusses the experimental aspects of the tritium NMR experiment. Especially useful for beginners is the explanation of sample preparation on p 18 and general safety on p 20 and 21. Chapter two provides a useful survey of tritiation methods, but its chief asset is the inclusion of tables listing for each tritiated compound details of the tritium NMR as well as the original literature reference. The final chapter reviews the application of tritium NMR spectroscopy to biosynthesis, catalytic studies, and organic reaction mechanisms. Of special interest to this reviewer was discussion of chirality imposed upon a molecule by tritium and its detection by tritium NMR as separate signals from a diastereotopic pair such as the case cited on p 175. We have recently observed this situation by tritium NMR for the 14-position of [14,15-^{3}H]dihydroforskolin. The volume concludes with a number of helpful appendices, over 200 original literature references, and a compound as well as subject index.

This book is well written and arranged. It answers all the questions that I have heard posed by beginners, about tritium NMR, presenting the technique as safe and uniquely informative. The volume is highly recommended for anyone interested in the subject of tritium NMR or the many special interests it so well serves.

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Advances in Drug Research. Volume 14. Edited by Bernard Testa. Academic Press, New York. 1985. ix + 339 pp. 15.5 × 23.5 cm. ISBN 0-12-013314-8. $69.50.

This is an excellent book. It covers four diverse topics, each well written. The largest chapter, Recent Advances in the Molecular Pharmacology of Benzodiazepine Receptors and in the Structure–Activity Relationships of their Agonists and Antagonists, occupies about half of this volume. It is an outstanding and encyclopedic treatment of the benzodiazepines which could only have come out of Hoffmann-La Roche. Even if one is not involved in this field, medicinal chemists and pharmacologists will be well rewarded by reading this work. The years of effort in this field provide a rare opportunity which the authors use to advantage to pursue structure–activity relationships and receptor-site developments, mechanism of action, and metabolic aspects in exquisite detail. Willy Haefely and three co-authors show excellent command of the material, which is well cross-referenced. Considerable previously unpublished activity data are presented.

Another extensive chapter, Drug Design in Three Dimensions, occupies about a third of the volume. It is written by N. Claude Cohen of Ciba-Geigy, Basel, from the perspective of one who has been using these approaches for many years. Monitor photos from the author's molecular modeling program, SCRIPT, illustrate the stepwise development of structural and electronic data. The concept of a fourth dimension in drug design, the inclusion of the electronic surface potential of a molecule, is discussed. Some of the eight color plates in this chapter apply to this aspect.

Stereochemical considerations in structure–activity relationships are illustrated in over a dozen drug classes. Approaches to receptor visualization in relation to known substrates, and other principles are developed first with critical analyses and discussions of conflicting interpretations and proposals. The concept of enzyme-excluded and enzyme-essential volumes is illustrated. The concluding section addresses the rational design of new leads, or ab initio drug design. Compelling examples should leave few doubters of the value of these approaches.

There is a chapter on Deuterium Isotope Effects in the Metabolism of Drugs and Xenobiotics: Implications for Drug Design, by Allan Foster of the Institute of Cancer Research in Surrey, England. It is a succinctly written coverage of the use of deuterium for the analysis of enzymatic reactions and as a tool to understand the mechanism of action of a drug or to identify the active species. "Metabolic switching" to alternate pathways as a consequence of deuteration is discussed. It is a limitation to the development of deuterated drugs. The magnitude of isotope effects for different functionality, and the distinction between intrinsic and observed effects are discussed. The effects of deuteration on binding, partition coefficients, and pK_a's are also covered.

The Mechanism of Action of Antiinflammatory Agents is the title of a chapter by William E. M. Lands of the University of Illinois at Chicago. He presents an interesting approach to the analysis of inflammation that has rarely been discussed. The focus of the analysis is on the rate-limiting step of the inflammatory process. The rate-limiting step, and therefore the appropriate treatment, may vary with the stage of development of the disease. The appropriateness of an experimental model depends on the rate-limiting step bearing some relation to the human situation. Many possible sites for intervention in the inflammatory process are discussed, but emphasis and detailed discussion are given to fatty acid oxygenase mechanisms. A strong case is made for the use of noncompetitive and reversible antioxidant inhibitors, particularly in the early stages where the tissue peroxide tone is low.

This book is remarkably free of errors. It is recommended reading for all medicinal chemists.

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Problems of Drug Dependence, 1983. Proceedings of the 45th Annual Scientific Meeting, The Committee on Problems of Drug Dependence, Inc. NIDA Research Monograph 49. National Institute on Drug Abuse, Rockville, MD. 1984. xii + 455 pp. 145 × 23 cm.

This volume contains both scientific papers and progress reports on the screening of new drugs for narcotic activity and addiction liability. There is considerable emphasis on behavioral testing in both animals and patients. Treatment programs that are evaluated include methadone maintenance, naltrexone therapy, and behavior modification.

Some of the brief sociological studies deal with the role of the social control on sex differences in drug abuse and criminality among heroin addicts. The latter reports a two-thirds drop in the crime rate of heroin addicts during periods when they were

drug free. Another interesting survey, based on interviews with callers to a cocaine "help line", summarizes physical and psychological problems encountered among cocaine addicts. These findings do not support the common misconception that cocaine is nonaddicting, especially when taken by the intranasal route.

Several contributions deal with practical problems, such as the abuse potential of a combination of pentazocine and tripelennamine (known on the street as "T's and blues"), and whether the mixed agonist–antagonist buprenorphine can be used to treat opioid dependence.

The growing appreciation of tobacco smoking as an addiction problem is reflected in papers on the oral behavior of smokers and on the use of the ganglion blocker mecamylamine to treat nicotine dependence.

Progress reports on the evaluation of new compounds are of little interest to the average reader. It would be most helpful to have a summary that highlights the unusual or key findings from the screening programs, such as new classes of narcotic drugs, compounds with unexpected specificity or potency, etc. Because dependence testing in monkeys is a unique feature of the screening program, it would be useful to point out those new compounds that are most likely to produce physical dependence.

On the other hand, the broad range of addiction-related topics surveyed here provides an overall view of the ways in which drug dependence is affecting both addicts and society. It also illustrates the kind of research that is being undertaken in order to understand basic mechanisms, as well as to achieve greater control of a glaring misuse of drugs that were originally developed to reduce human suffering.

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Organosphosphorus Chemistry. Volume 15. Edited by D. W. Hutchinson and B. J. Walker. The Royal Society of Chemistry, London. 1985. xiv + 335 pp. 14 × 23 cm. $122.00.

This volume is similar to past volumes except that there is a review, "Phosphazenes", by J. C. van de Grampel and B. de Ruiter. There is a particularly strong section, about 60 pages, on "Nucleotides and Nucleic Acids", by J. B. Hobbs, which covers the rapidly developing advances in synthesis in this area. This review also includes sections on sequencing and on analytical methods. The remaining sections of the volume, including the authors, represent continuations of the valuable contributions that have been demonstrated in past volumes.

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Books of Interest

Advances in Enzymology. Edited by Alton Meister. Wiley, New York. 1985. v + 513 pp. 16 × 23.5 cm. ISBM 0471-89011-1. $55.00.

Understanding Enzymes. Second Edition. Edited by Trevor Palmer. Halsted Press (a division of Wiley), New York. 1985. 411 pp. 16 × 23.5 cm. ISBN 0470-20173-8. $45.00.

The Third Dimension in Organic Chemistry. Wiley, New York. 1985. Alan Bassindale. xiii + 242 pp. 14 × 23 cm. ISBN 0-471-90189-X.