JOURNAL OF MAGNETIC RESONANCE **125,** 228–230 (1997)
ARTICLE NO. MN971105

BOOK REVIEWS

NMR as a Structural Tool for Macromolecules. Current Status and Future Directions. B. D. Nageswara Rao and Marvin D. Kemple, Eds. Plenum, New York, 1996, 382 pages, $120.00. ISBN: 0-306-45313-4.

The scope of ''Macromolecules'' in the title of this book is limited to naturally occurring species. A symposium on the topic of the title was held in 1994 in Indianapolis, Indiana. The list of contributors included many of the outstanding names in NMR investigations of peptide, protein, and nucleic acid structures, as well as in macromolecular dynamics. To name some of them: Ernst, Wagner, Kay and Forman-Kay, Chazin, Bax, Redfield, Kumar, Guéron, Kaptein, James, Markley, Fesik, Clore and Gronenborn, Wright, and Sykes.

The 22 chapters in this volume represent somewhat expanded writeups of the talks presented at the symposium; also included are reproductions of the abstracts of some 70 posters. Most of the lectures represent summaries of recent work by the contributors, including extensive references to published papers. What is said in the bodies of these contributions about ''Future Directions'' is rather limited; emphasis is rather on ''Current Status.'' This reviewer, in truth, found the most interesting parts of the volume to be the verbatim transcripts of discussions following the talks as well as of two formal panel discussions; the topics of the latter are extensions to larger molecules and comparisons with other structural methods.

The book gives a very good portrayal of the state of the art in 1994, and would be helpful to an experienced NMR spectroscopist seeking examples to follow in setting up an experimental program in this area. Although the volume was produced by photographic reproduction of manuscripts by the several contributors, which differ in type face, layout, and form of reference, the reproduction is excellent and the text is quite readable.

W.S.B.

Nonlinear Computer Modeling of Chemical and Biochemical Data. James F. Rusling and Thomas F. Kumosinski. Academic Press, San Diego, 1996, 268 pages, $64.95. ISBN: 0-12-604490-2.

The mathematics of linear and nonlinear regression analysis and applications of these methods to the fitting of data from various types of experimental measurements, mostly spectroscopic, are described. There is one fairly short chapter on analysis of NMR relaxation, including that of ^{2}H, ^{17}O, and ^{1}H nuclei in protein solutions and of ^{13}C nuclei in the solid state.

W.S.B.

Solving Problems with NMR Spectroscopy. Atta-ur-Rahman and Muhammad Iqbal Choudhary. Academic Press, San Diego, 1995, 430 pages, $34.95 (paper). ISBN: 0-12-066320-1.

The authors state that ''this book is intended to provide practical knowledge to research workers in the use of NMR spectroscopic techniques to elucidate the structure of organic molecules.'' The first chapter is a general introduction to pulse NMR, and the second chapter describes the two essential components of complex experiments, the formation of spin echoes and polarization transfer. Chapter 3 describes the considerations involved in setting up 2D experiments and processing the data from these, and Chapter 4 is a detailed consideration of the nuclear Overhauser effect. Chapter 5, by far the longest, contains a detailed presentation of the procedures and pulse sequences for the various common experiments used for resolving overlap or correlating resonances, along with examples of spectra obtained by these sequences.

The unusual feature of the book, reflected in the title, is the large number of ''problems,'' interspersed throughout the text of the first five chapters, with solutions to the problems given at the end of each chapter. A fair number of these problems, especially in the earlier chapters, merely call for the answerer to repeat or summarize what has been stated in the text and are thus rather trivial. When the stage of assigning resonances in spectra or predicting the behavior of nuclei in response to pulses is reached, the problems become quite useful as a learning tool. Some numerical calculations, such as obtaining a value of T_1 from inversion-recovery data or an RF power level from off-resonance decoupling, could certainly have been added with profit.

Chapter 6 gives a short presentation of 3D experiments, with emphasis on the types of cross peaks obtained. Chapter 7 describes recently developed methods using ''soft'' pulses as well as the use of field gradients. Finally, Chapter 8 includes detailed presentations of the logical procedures—an 11-step formal sequence is provided—which were used in determining the structures of two fairly complex natural-

1090-7807/97 $25.00

product molecules. The first of these examples is instructive, but the second does not even show all the spectra that are mentioned and is too rushed to permit the reader to follow the arguments readily.

On the whole, this book should be quite useful for someone who has at least a nodding acquaintance with the basics of NMR and wishes to develop a better understanding of pulsed experiments and the ways in which results obtained from them can be analyzed. To illustrate some irregularities in the presentation, I shall mention just one point. On reading p. 66 (still in the first chapter), one learns that composite pulses have been used in broadband decoupling experiments. On p. 98 (in Chapter 2): ‘‘The decoupler is off during the first $1/J$ delay period.’’ Since the reviewer could not recall what had been said earlier about the ‘‘decoupler,’’ decoupling, or double resonance, he consulted the index and then the ‘‘Contents’’ but found no relevant entry in either one. Only someone with preliminary acquaintance with NMR spectroscopy would be able to understand these allusions to decoupling or to follow all the content of this otherwise useful book.

W.S.B.

Multidimensional Solid-State NMR and Polymers, Klaus Schmidt-Rohr and Hans Wolfgang Spiess. Academic Press, London, 1994, 470 pp., £65.00/$99.95. ISBN: 0-12-626630-1.

Solid-state NMR spectroscopy has seen amazing progress in the past few years. A portion of the development has come directly in relation to novel ways of studying and interpreting dipolar interactions and their anisotropic aspects, but a substantial portion of the progress owes much to the transfer of ideas from liquid-phase NMR. This volume weaves together the two areas and presents in detail the physics and mathematics on which solid-state NMR is based. Much of this theoretical basis is, of course, also applicable to liquid-phase NMR. The applications described in this volume are to synthetic polymers with emphasis on measurements of dynamics and of order. Results are provided as examples of the use of various methods, rather than in the form of a systematic review of polymer behavior.

In the introductory chapter, several fairly simple illustrations are given in order to show some of the types of information which can be obtained by NMR. Chapter 2, ‘‘Principles of NMR of Organic Solids,’’ contains a detailed quantum-mechanical treatment of NMR in terms of the relevant Hamiltonians, the equation of motion of the density operator, and the effects of RF pulses. Chapter 3, ‘‘High-Resolution NMR Techniques for Solids,’’ extends the theory to various aspects of pulse sequences used for solids, including such aspects as the analysis of the sequences in terms of average Hamiltonian theory. Also considered are cross polarization, spin diffusion, selective excitation, magic-angle spinning and side bands, second-order quadrupolar effects, multiple-quantum coherence, zero-field experiments, and relaxation.

A generally useful description of Fourier analysis is the subject of the next chapter, including data-sampling considerations, signal-to-noise ratio, and processing of 2D data. This is followed by a very brief description of polymer structure and terminology, as a concession to the uninitiated. Chapter 6 begins with concentration on 2D experiments, as applied to solids, for purposes of separation of parameters or of correlation between various spectra of a system, a distinction which, the authors stress, is often arbitrary, especially for solid-state NMR.

Chapters 7, 8, and 9 are devoted to methods for studying the details of polymer dynamics by multidimensional exchange experiments. In Chapter 10, experimental aspects of pulse sequences for multidimensional exchange spectroscopy and details of processing of data from these sequences are presented. Chapter 11 is devoted to simulation of these spectra, based on various models. In the last two chapters, attention is turned from the main theme, polymer dynamics, to polymer structure: order, domain size, and internuclear distance. The book ends with 25 pages of appendices covering mathematics related to the content of the main text.

This volume is a comprehensive treatment of the theory and methodology of solid-state NMR, particularly those aspects related to the study of motion and structure in synthetic polymers. It is not easy to read, for the mathematics often comes pretty fast. It is frequently difficult to find the forest for the trees. However, any NMR spectroscopist working in this area or planning to study polymers should find it extremely useful.

W.S.B.

NMR Spectroscopy Techniques, 2nd ed., Martha D. Bruch, Editor. Dekker, New York, 1996, 616 pp., $195.00. ISBN: 0-8247-9450-8.

This is Vol. 21 in the ‘‘Practical Spectroscopy’’ Series, edited by Edward G. Brame. It comprises 10 chapters of varying length, 3 of which are authored or coauthored by the editor of the volume. The book begins with a general introduction by Traficante to NMR and how a spectrometer works. Next is a chapter by Bruch and Dybowski on ‘‘Spectral Editing Methods for Structure Determinations,’’ in which are discussed methods for correlating multiplets by double-resonance methods, including decoupling and observation of the NOE, as well as methods for solvent suppression.

‘‘NMR Relaxation and Dynamics,’’ by Wasylishen, gives an excellent introduction to the meaning of the several relaxation parameters and techniques for their measurement. Also included are equations for relaxation in terms of transition probabilities and a discussion of the mechanisms contributing to relaxation. Next comes ''Multidimensional NMR Spectroscopy of Liquids,'' by Bruch. Following a general introduction to the 2D idea, there is fairly extensive presention of *J*-resolved spectroscopy. Correlation methods are illustrated with COSY, RELAY, TOCSY, and HETCOR experiments. There is a bit about indirect detection. Exchange spectroscopy, in the form of NOESY and ROESY methods, is described, and 3D and 4D methods are very briefly sketched. ''Small Organic Molecules: Practical Tips and Structure Elucidation'' includes some examples of use of chemical shifts and coupling constants for establishing structures, along with consideration of chiral effects, shift reagents, and reaction rate measurements.

''Structure Determination of Biological Macromolecules,'' by Rizo and Bruch, is the longest, most detailed, and perhaps the most valuable chapter in the book. As for many of the other chapters, there is an extensive series of references. Peptides, proteins, oligosaccharides, and other carbohydrates are all considered, with specific examples of the methods of interpreting data from various experiments. For proteins, methods for sequential assignments and for determination of conformation and of secondary structure are included.

For spectroscopy of solids, there are two chapters. That by Jelinsky and Melchior is devoted primarily to the ''high-resolution'' observation of carbon-13 in a proton-containing solid. Experimental aspects of magic-angle spinning and of several experiments in which MAS is a part are discussed. A chapter by Dybowski is concerned with the observation of ^{1}H, ^{19}F, and ^{2}H, both in the form of ''wide-line'' spectra and with some of the effects of the various nuclear interactions, for which the theory is developed, removed by pulse sequences of the WAHUHA type, including CRAMPS.

Chapter 9, ''Chemical and Physical Characterization of Polymer Systems by NMR Spectroscopy,'' by Brandolini introduces sequential and tactivity aspects of polymer structure, and illustrates the NMR study of these by both liquid-state and solid-state spectra. This section is too short to cover much ground. Chapter 10, ''In Vivo ^{31}P and ^{23}Na Spectroscopy and Imaging,'' by Seshan and Bansal, is interesting and instructive, but seems out of place in this book.

The book is nicely produced and carefully edited. There are extensive lists of references. However, this reviewer found a few problems. There is no list of contents which would guide the reader to the many sections and subsections into which each chapter is divided. Adding to the difficulty of finding the location of a specific topic is the rather sketchy nature of the index. As examples, there are no entries for ''Carbon-13'' or for ''CRAMPS,'' subjects for which I happened to look in preparing the review. There is at least one oversimplification to which many purists would object. On page 192: ''. . . the mixing pulse in the COSY sequence creates multiple-quantum coherence, which is transverse magnetization. . . .''

This book might have a subtitle such as ''How and where they should be used,'' or perhaps ''Practical aspects of the experiments.'' Were it not for the amazing list price, the book could be recommended as a very readable and useful introduction to NMR spectroscopic experiments with only a minimum of complex theory and mathematics.

W.S.B.