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Book reviews

^{13}C-N.m.r. Spectroscopy: A Working Manual with Exercises, by EBERHARD BREITMAIER AND GERHARD BAUER, translated from the German by BRUCE K. CASSELS, Harwood Academic Publishers, New York, 1984, x + 356 pages, $108.00; 10 or more copies (textbooks) $44.95 each.

This book was originally published in German in 1977 as *^{13}C-N.m.r. Spektroskopie: Eine Arbeitsanleitung mit Übungen* by Georg Thieme Verlag. It is unclear why the secondary publisher has taken so long to produce an English translation. In such a rapidly advancing subject as n.m.r. spectroscopy, this delay guarantees that the treatment will be less up to date than one could wish. Such important, modern spectral-assignment techniques as two-dimensional n.m.r., determination of carbon–carbon connectivity by double quantum methods, and spectral editing by polarization transfer and multiple quantum methods are not discussed.

The book has been published as volume 3 of the "MMI Press Polymer Monograph Series", a series that purports to present international accounts of research and developments in the specialized areas of macromolecular chemistry, but the connection of this volume with research on polymers is tenuous, at most. Polymers are mentioned at only two locations in the book, the rest of the text being devoted to n.m.r. techniques and organic molecules of low molecular weight, including 12 carbohydrates or derivatives thereof. In the rather brief Subject Index, which is about three pages in length, there is no mention either of polymers or macromolecules.

The text is arranged in two parts. Part I (104 pages) is comprised of nine chapters of general discussion of ^{13}C-n.m.r. techniques, and methods for spectral interpretation. Part II is subdivided into Section A: – "Exercises in the Interpretation of ^{13}C-N.m.r. Spectra and Their Application to Structural Problems in Organic Chemistry" (pp. 105–264), and Section B: – "Solutions to the Problems in the Interpretation of ^{13}C-N.m.r. Spectra" (pp. 266–344). Additionally, there is a somewhat inadequate bibliography (less than one page), and appendices that contain lists of chemical substances and compounds discussed in the text and the exercises.

Chapter 1, "Basic Principles and Quantities of Nuclear Magnetic Resonance Spectroscopy", presents a brief, non-mathematical introduction to the n.m.r. phenomenon, and the concept of spin–lattice relaxation is introduced.

Chapter 2, "Measurement of ^{13}C-N.m.r. Spectra", explains the pulsed n.m.r. experiment, the pulse-Fourier-transform method, and the use of smoothing functions. The approximate range of spectral excitation by the radiofrequency pulse is given incorrectly on p. 8; it should be $\nu_1 \pm 1/t_p$.

Chapter 3, "Proton Decoupling in ^{13}C-N.m.r. Spectroscopy", details the need for this technique in ^{13}C-n.m.r. spectroscopy, and the various conventional ways in which it can be used, including the powerful, graphical off-resonance method for correlation of ^{1}H and ^{13}C chemical shifts. The use of the term "pulsed proton decoupling" for techniques that are often known as "gated" or "inverse gated" decoupling now seems confusing, in view of the recent development of a different technique termed "composite pulse, proton decoupling".

Chapter 4, "Chemical Methods of Signal Assignment", briefly describes the use of isotopic labeling with ^{2}H, ^{12}C, or ^{13}C isotopes, and also the use of paramagnetic, chemical-shift reagents.

Chapter 5, "Sample Concentration: Solvents and Standards", could logically have been placed nearer to the beginning of the book.

Chapter 6, "^{13}C Chemical Shifts: A Survey", briefly outlines the dependence of ^{13}C chemical shifts on organic structure.

Chapter 7, "Interpretation of ^{13}C Chemical Shifts", offers a much more comprehensive discussion than do the earlier chapters, and substantial attention is given to the increments in chemical shifts generated by various classes of substituents, and by configurational influences in saturated, six-membered rings.

Chapter 8, "Analysis of Coupling Constants", describes the structural significance of carbon–proton, carbon–carbon, and carbon–heteroatom, nuclear coupling-constants, but with only very limited reference to the Karplus relationship with dihedral angle. A number of useful tables of data are included in this chapter.

Chapter 9, "^{13}C Spin–Lattice Relaxation Times: Measurement and Range of Application", presents a good discussion of several pulse techniques for measurement of spin–lattice relaxation times (T_1), and the relationship between molecular motion and dipole–dipole relaxation is nicely explained.

The foregoing chapters contain a number of attractive qualitative explanations of physical phenomena that should quite painlessly increase the reader's understanding of the subject. The translation is very good, but the reader needs to beware of a number of errors. Treatment of the nuclear Overhauser effect (n.O.e., a subject of perennial confusion) is weak in this book, throughout which it is not made clear that the ratio of the intensities of enhanced and unenhanced ^{13}C-n.m.r. signals is ~3 and *not* 2. For example, on p. 16, it is stated incorrectly that a CH doublet with relative intensity ratio 1:1 becomes, on decoupling, a singlet having intensity 4.

In the early part of the book, there are several errors in the captions of the Figures. The captions for Fig. 2.1 are confused, and those for Figs. 3.1 and 3.2 should have the labels B and D, and A and B exchanged, respectively. It seems quite unlikely that the ^{13}C-n.m.r. spectrum shown in Fig. 3.4 was recorded at 10 MHz. The spectra shown in Figs. 3.2, 3.3, 3.4A, 3.5(B–G), and 3.7 have no units on the chemical-shift scale. Fig. 6.2 has two captions, because of duplication of the caption for Fig. 6.3.

On p. 37, it is stated that the recommended volume of solution for ^{13}C

measurements is about 1 mL. This is acceptable for 10-mm sample-tubes, but this volume is certainly not optimal for such other sample-tube sizes as 5, 15, 20, or 25 mm.

Part II, Section A consists of 76 problems in the analysis and interpretation of ^{13}C-n.m.r.-spectral data. Each problem is concisely stated in bold-face type, and the recording conditions for the spectra are also given, usually in a total space of much less than one page. Depending on one's point of view, the blank space on each page may be considered to be either wasted space, or work space, albeit expensive. The spectra are printed on the facing pages, either horizontally, or, in many cases, side-ways, in a less convenient orientation. The spectral data are clearly reproduced, and they have been collected from a wide variety of organic molecules of low molecular weight. Usually, a proton–noise-decoupled spectrum is given, together with either an off-resonance-decoupled spectrum, or a proton-coupled spectrum. The ^{13}C-n.m.r.-spectral data were measured at frequencies of 15.08, 20, or 22.63 MHz, values that lie somewhat below the frequency range of current research spectrometers. The application of T_1 measurements, variable-temperature techniques, and shift reagents is included in the set of problems. Despite being somewhat out of date, these exercises could still provide the reader with many hours of stimulating mental activity. However, only three exercises on carbohydrate derivatives have been included.

Solutions to the problems are discussed in detail in Section B, and considerable attention is given to analysis of the data in terms of empirical, chemical-shift increments. In fact, the reader may be surprised at just how far this type of data analysis can be pursued. The text contains a number of minor errors in carbohydrate nomenclature; for example, on pp. 34, 334, 335, 340, 341, and 342. The Figures, formulas, and Tables are uniformly good.

Because this text is somewhat out of date and because of the high cost of individual copies, it cannot be recommended as being cost-effective for the personal library of the professional scientist. At this point in time, this volume appears to be more suitable as a textbook for a course that would introduce students to conventional ^{13}C-n.m.r.-spectral assignment-techniques. In general, the approach adopted is less comprehensive than that of an earlier text, *Interpretation of Carbon-13 N.m.r. Spectra* by F. W. Wehrli and T. Wirthlin, Heyden, London, 1976. The latter volume also contains a set of assignment problems (30), with solutions.

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New Approaches to Research on Cereal Carbohydrates: edited by ROBERT D. HILL AND LARS MUNCK, Elsevier, Amsterdam, 1985, xii + 415 pages, $101.75, Dfl. 275.00.

This book constitutes Volume 1 of the series title, namely, *Progress in Biotechnology*. This volume is a collection of 47 papers in the form of Proceeding of the International Conference on New Approaches to Research on Cereal Carbohydrates, held in Copenhagen, Denmark, June 24–29, 1984, under the chairmanship of Professor Roy L. Whistler. The subject area is wide, but appropriate to the title of the book, and covers cereal research, molecular biology, genetic variations, enzymology, fundamental carbohydrate chemistry, and topics of technological importance, including nutrition. The papers are grouped under six sections.

The first section, entitled Starch Chemistry, which should have been designated "Starch Chemistry and Biochemistry", has 18 papers, of which 6 are excellent reviews. The latter deal mainly with the fine structure of starch, enzymes used for structural studies, enzymic and genetic control of starch biosynthesis, and synthesis and regulation of starch-hydrolyzing enzymes. The other papers deal with the examination of minor components of starch, including lipids; genetic control of properties of starch and its synthesis; regulation of alpha amylase synthesis; starch–lipid complexes, and action of alpha amylase on such complexes; mechanism of action of alpha amylase; physical studies on starch in solution, and its pasting and gelling properties; and finally, starch retrogression. Thus, this section provides a reader with a good deal of biochemical information about cereal starch. The references cited in the reviews are of additional importance to a researcher.

The second section, Starch Technology, has two reviews, both dealing with the use of starch in the plastics industry. There are three research papers: extraction of starch; ethanol production from starch; and possible industrial application of a unique, fungal, acid-stable alpha amylase. This section is rather thin in substance, and, as the book deals with biotechnological progress, this reviewer was looking for a greater number of contribution on this topic.

The third section is devoted to the Chemistry of Non-starch Polysaccharides. Two important reviews are on hemicelluloses of grasses and on cellulose biosynthesis. The research papers include the chemistry of cell-wall β-D-glucans and glucuronoarabinoxylans; wheat pentosans; and various carbohydrate fractions of cereal straw. There are two miscellaneous, but interesting articles, namely, on the use of lectins in cytohistochemical study of grains, and dye–polysaccharide interaction. What are missing are papers on structures, and inter-relations, of cell-wall polysaccharides.

The fourth section is on Non-starch Polysaccharide Technology. There are three reviews in this section, the subjects of which are enzymic hydrolysis of cellulose; straw, and wood cellulose for paper-making; and industrial utilization of cereal plants. Of the three other research papers in this section, two deal with

cellulose hydrolysis, and the third with digestion of ammonia-treated straw by ruminants. Thus, the overall coverage is quite limited.

The fifth section is on Nutrition, with two reviews: cereal carbohydrates for humans, and grains and straw for ruminants. Three other papers describe food aspects of cereals, including the nutritional value of the aleurone layer of cell walls; cereal-based carbohydrates; and grain β-D-glucan for chickens. The fourth paper is on g.l.c. analysis of non-starch polysaccharides, and should have been placed in the third section.

In the sixth, and final, section Prof. R. L. Whistler sums up developments in cereal chemistry, and gives general comments on future research. There is also a summary, by Dr. J. H. Looker, on the teaching of Carbohydrate Chemistry in Universities.

This reviewer noted some mistakes and inconsistencies in this book, which presumably arose from the type of book production, where the author had no opportunity to see the article in proof form. For example, *p. 1*, the terms synthase and synthetase have been used, but the former is now generally accepted; *pp. 3 and 4, Vicia fabia* should read *Vicia faba; p. 7, line 1,* pyrophosphates should read pyrophosphatase; *p. 9, Table 2,* for what does N.D. stand — not determined, or not detected?; *p. 14,* horesradish should read horseradish; *p. 20*, Roman numerals II, III, and IV are shown as paragraph headings, but there is no I; *p. 21*, under *Problems,* there is I, but there are no subsequent numbers; *p. 35, Fig. 1,* the nature of the chromatography used is not described; *pp. 122, 124, 126, Figs. 1, 4, 8:* is the brace sign { meant for μ?; *p. 127, line 20*, the reference 14 in brackets should be 15; *p. 239, under ref. 7,* four different papers are cited, and the year of publication is missing from one of these. The absence of an Index at the end of the book is painfully noted.

On the whole, this book is unique, timely, and in keeping with the rapid growth in research on biotechnology. The readership of this book should include analytical chemists; biochemists; agricultural, organic, polymer, and physical chemists; genetic engineers; enzymologists; and food technologists.

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