

Chimie des Substances Naturelles (Paris). The authors will be from various countries, but with some exceptions, publication will be in French.

The English-speaking chemist who refers to the lucid pages of "Les Cyclitols" will be pleasantly reminded that scientific French is the easiest of foreign languages to read. Now that an increasing number of valuable French scientific works are reaching us, it is hoped that no imagined language problem will prevent the American chemist from making full use of them.

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Developments in Inorganic Polymer Chemistry. Edited by M. F. LAPPERT, Senior Lecturer in Chemistry, and G. J. LEIGH, Lecturer in Chemistry, both at the Manchester College of Science and Technology, University of Manchester (England). American Elsevier Publishing Company, Inc., 52 Vanderbilt Avenue, New York 17, N. Y. 1962. xi + 305 pp. 14 × 22 cm. Price, \$10.00.

The knowledge that inorganic compounds can combine into polymeric substances is not new, but until ten years ago the subject received scant attention. Recently, however, there has been a great deal of interest in it, and a voluminous literature on inorganic polymers is building up. Thus, this book is the third to appear within the last year and a half. All three are quite different, and all deserve attention, for not only is the chemistry involved very interesting, but there is reason to believe that practical results are not far in the future.

This volume records a series of lectures which were given at the Manchester College of Science and Technology in the fall of 1960. It is unfortunate that publication was delayed for two years, because in a field which is growing as rapidly as this one, many changes take place in that length of time. However, in spite of the delay, the information in these lectures, and the compilations of the hundreds of references which accompany them, are of great value.

Following a short chapter which serves as a general survey of the field, there are chapters on polymers containing boron and nitrogen, other boron compounds, phosphorus and nitrogen, sulfur and phosphorus, silicones, siloxanes (two chapters), and metal chelates. The last is the shortest chapter in the book, covering only seventeen pages. The reviewer feels that the author of that chapter is not as hopeful as he should be about the future of coordination polymers, though he agrees "that the difficulties are much greater than was foreseen." In general, the chapters are descriptive, and give little information on factors that influence polymer formation and thermal stability. Most of the reviews are reasonably complete, and in some cases, critical. The quality of the writing is good, and is more nearly uniform than in most books which have been prepared by a number of authors. Readability could have been improved by tabulating some of the information and by the use of formulas of compounds instead of their names; e.g., bis-(triphenylsiloxy)-diisopropoxytitanium, which few chemists will visualize without puzzling over it or writing the formula.

Because of the mass of detailed information which it contains, this volume is not easy to read for general background information. For the same reason, however, it will be highly useful to those interested in inorganic polymers and as a reference book on the chemistry of the non-metals.

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Mass Spectrometry. Organic Chemical Applications. By KLAUS BIEMANN, Associate Professor of Chemistry, Massachusetts Institute of Technology. McGraw-Hill Book Company, Inc., 330 West 42nd Street, New York 36, N. Y. 1962. xii + 370 pp. 16 × 23.5 cm. Price, \$13.75.

Since mass spectrometry is by no means a new physical tool nor does the field suffer from a dearth of recent books or review articles, the present opus should be reviewed in the light of the current status of the subject and its monograph literature. It is interesting to note that while mass spectrometry has been practiced in certain areas of organic chemistry (notably the petroleum field) for nearly twenty years, it has only recently begun to enter the every-day organic laboratory. This is not the fault of instrumentation—the usual reason for the lag between the development of a physical tool and its extensive use by the laboratory organic chemist—since some of the presently employed mass spectrometers have been available for many years. It cannot even be blamed on the relative non-volatility of the bulk of organic substances, because O'Neal and Wier introduced a heated inlet system over ten years ago.

In my opinion, the chief reason for this situation is implicit in a sentence taken from Biemann's introduction, namely "... that the most useful data are obtained if the originator of the problem and the person determining the spectrum are in close contact. The most ideal situation results if both the chemistry and the mass spectrometry required for the solution of a given problem are carried out by the same individual..." In other words, with few exceptions—most of them rather recent ones in Sweden, Austria, Australia, Great Britain and the U. S.—the bulk of mass spectrometric research was not performed by organic chemists. A second reason, again in the reviewer's opinion, is that much of the mass spectrometric information accumulated in the past has been published in journals, not generally read by organic chemists, and presented in a manner which was hardly likely to excite the reader. Some of the striking exceptions to this generalization, such as McLafferty, Stenhagen and Biemann, only seem to confirm its correctness. Fortunately, this state of affairs is changing rapidly and the present book is likely to accelerate the process. It is not the first book dealing with mass spectrometric applications to organic chemistry, but it is the first written by an organic chemist and for his purposes by far the best.

The first chapter, dealing with instrumentation, gives a brief description of the different mass spectrometers. As the author points out, fuller details can be found in other books dealing with mass spectrometry, but in my opinion it would have been very desirable for the average organic chemist if the various instruments were identified with existing commercial models (U. S.: CEC and Bendix; Great Britain: A.E.I.; Germany: Atlas; Japan: Hitachi) so as to help him in the potential choice of an instrument for his laboratory. Even more important would have been an authoritative comment, addressed to instrument companies, reminding them that the ideal mass spectrometer for general organic chemical work still needs to be produced.

Chapter 2 on "Sample Handling and Operating Techniques" is particularly valuable to the novice in the field and illustrates the various procedures practiced by the author in his own laboratory. Most readers will find that eventually each mass spectroscopist develops his own favorite methods, especially suited to his needs. Thus the reviewer's laboratory would take issue with Biemann's conclusion on p. 23 that the most valuable means of handling a "non-volatile" substance is to convert it into a more volatile derivative. Before attempting this, we would always examine first the direct insertion of the sample near the ion source, especially since this is now possible with mass spectrometers showing unit resolution to masses beyond 800. In the section (p. 42) on the presentation of data, Biemann favors figures containing plots of relative abundance *vs.* *m/e* over the historically more standard tabular presentations found especially in journals such as *Anal. Chem.* In the reviewer's opinion, this point cannot be made too strongly, because as far as the organic chemist is concerned, no other factor has made the reading of many mass spectrometry papers so unpalatable an experience.

The third chapter on "The Nature of Mass Spectra and Their Interpretation" is by far the longest. It commences with a very good discussion of the recognition of molecular ions, the first problem usually encountered by the organic chemist. There follows a detailed, and on the whole very clear, coverage of fragmentation mechanisms, utilizing the approach first pioneered by McLafferty and reviewed by him in an able manner in a recent chapter (Chapter 2 in "Determination of Organic Structures by Physical Methods," F. C. Nachod and W. D. Phillips, Ed., Academic Press, Inc., New York, N. Y., 1962), which may well make good introductory reading before delving into Biemann's book. This section abounds with mechanisms and thus represents the diametric opposite of Beynon's presentation of mass spectral fragmentation processes ("Mass Spectrometry and its Applications to Organic Chemistry," Elsevier Press, Amsterdam, 1960). If I would have to choose between these two extremes, I would certainly pick Biemann's exposition and expansion of McLafferty's views, but I would feel much more comfortable if many of the mechanisms were put forward in a more conditional manner. The very positive statements using mechanisms A, D, H, etc., represent a very tempting siren's song, which will lull the average organic chemist into a comfortable feeling of mechanistic security, which is quite unreal and unjustified at this stage of our knowledge.

While several fragmentations, notably the simpler ones, have been substantiated by deuterium labeling (e.g., p. 113), there are others (the vast majority) for which there exists as yet no verification. As far as possible, mechanisms should be supported by labeling experiments, and while this sermon is preached elsewhere in the book, it is not practiced very extensively in this section. In the absence of such support, it may still be preferable to suggest possible mechanisms for major fragmentations, rather than to use the earlier mass spectroscopist's proverbial wiggly line, but these mechanisms should be proposed cautiously and not *ex cathedra*. This is particularly important in a book of this kind, which is bound to influence the modern graduate student because it appeals to his desire for sophistication. As an example, an experimentally unsupported hydrogen transfer

mechanism for a tricyclic acetal (p. 119) is presented in the following terms, the capital letters referring to standard mechanisms employed by Biemann throughout the book:

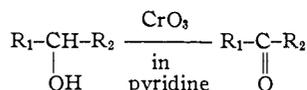
"The loss of the cyclooctyl group from the acetal carbon atoms corresponds to Type B, the resonance form of which undergoes intramolecular hydrogen migration (Type G), which in the case of a cyclic ion does not result in fragmentation. It takes place in the last step, which constitutes merely an electron shift, resulting in elimination of a molecule formic acid and formation of the allylic cation of mass 123."

This is pretty potent and tempting stuff for a graduate student or uncritical reader and could well be diluted with a few "may" or "might" or "possibly," in the absence of confirmatory evidence. Thus, the transfer of a γ -hydrogen to oxygen, originally proposed by McLafferty and subsequently established in esters through deuteration experiments by Stenhagen and by Dieckman (in Biemann's Laboratory), is stated categorically on p. 122 to apply also to acids, aldehydes, olefins, alkylbenzenes, phenylethanol, amides, nitriles, etc. While such hydrogen transfers are plausible and mechanistically very attractive, only very little proof has as yet appeared in the literature, Meyerson's deuterated propylbenzene being a convincing example. Obviously, more caution must be exercised and, even more importantly, more experimental work must be done with suitably labeled substrates before such hydrogen transfer mechanisms can be employed routinely without reservation.

The final section of this chapter contains a clear and succinct discussion of the important metastable peaks (section 3-3). Especially gratifying is the direct reproduction of a mass spectral photographic record containing such a metastable peak; organic chemists getting all of their mass spectrometry exposure from reading journal articles are unlikely to visualize the appearance of such peaks, even though they are aware of their mechanistic significance. The discussion of negative ions (section 3-5), though short, is very much to the point and the advantages of positive ion mass spectrometry for the organic chemist are presented forcefully and convincingly. Admittedly, additional work is indicated in this rather neglected area of mass spectrometry.

Chapter 4 ("Additional Techniques") contains the type of information which is especially useful to the beginning practicing mass spectroscopist—an example being the detection of common laboratory contaminants in samples such as silicone or hydrocarbon grease, or pump oil. The average organic chemist will be amazed how often this occurs and it will be hardly a consolation to learn that the mass spectroscopist himself is sometimes responsible for such impurities arising from stopcock grease in certain inlet systems.

While some of the discussion in this chapter is quite sophisticated, certain sections dealing with "chemical pretreatment" cover completely elementary information. Thus (p. 178), "... carboxyl groups are quantitatively esterified (by diazomethane), a useful reaction (Eq. (4-8)) discussed later." This freshman chemistry approach applies to a number of reactions, illustrated on p. 180 by equations such as $\text{RCOOH} \rightarrow \text{RCOCl} \rightarrow \text{RCOOR}$ or by statements (in connection with the recovery of acids or bases from their salts) such as "... extraction of the acid with an organic solvent after acidification of an aqueous solution of the salt, or by a similar process for the isolation of the base." If this type of knowledge on the part of the reader cannot absolutely be taken for granted, then it is unlikely that he has survived reading the book to p. 180 or that he will be able to continue beyond this stage. At times, this type of advice is not only redundant, but not even optimal. On p. 190, in discussing the low abundance of molecular ions of secondary alcohols and the desirability of determining the mass spectrum on the derived ketone, it is stated simply that "oxidation can easily be achieved by treatment with chromic acid in pyridine," the reaction then being illustrated by



While the illustration is both correct and pointless (considering the level of the reader), it is debatable whether chromium trioxide in pyridine is the ideal or universal reagent for such small-scale experiments. The reviewer, if pressed in public to limit his choice to one reagent, would certainly suggest that the Jones reagent (chromium trioxide-sulfuric acid in acetone) is much more widely applicable and much more convenient on a small scale. On the other hand, the information given immediately below (pp. 190-191) that dinitrobenzoate derivatives of alcohols are quite volatile and yield useful mass spectra (illustrated in Fig. 4-9) is obviously pertinent and probably not so generally known.

The recommendation of p. 192 about the combined use of mass spectrometry and thin-layer chromatography should have been accompanied by a reference to Heyns in *Angew. Chem.*, **74**, 387 (1962), where many of these points were first covered. The

discussion of high-resolution mass spectrometry (section 4-6) is quite short and the reader is best referred to the book (*loc. cit.*) by Beynon, who pioneered the application of this technique to organic chemistry. The measurements of ionization and appearance potentials is covered only very superficially, properly so, since this subject does not contribute greatly at this stage to the type of organic chemical applications emphasized in the book. These potentials are obviously of mechanistic significance and are covered in substantial detail in Beynon's (*loc. cit.*) and Reed's ("Ion Production by Electron Impact," Academic Press, Inc., New York, N.Y., 1962, and reviewed by Stevenson in *J. Am. Chem. Soc.*, **85**, 248 (1963)) books, to name but two recent reviews.

Chapter 5 ("The Mass Spectra of Isotopically Labeled Molecules") contains some of the best sections in the book in its coverage of sources of error and calculations of isotopic species. Similarly, the various examples of applications of such isotope labels (chiefly deuterium and O^{18}) to various organic and biochemical problems are very well chosen. In the reviewer's opinion, it is unfortunate that the last section (5-6) "Applications in the Investigation of Electron-Impact Mechanisms" is only one page long and that the relevant material is scattered throughout the book, where it is not found easily. Surely, it would have been appropriate to place here in detail (and including the often rather difficult synthetic approaches to such labeled analogs) a few selected classical examples such as Meyerson's tropylium work or Stenhagen and Ryhage's extensive labeling of fatty acids—examples which are mentioned only briefly elsewhere (pp. 85 and 120) in this book.

The next chapter dealing with "Fatty Acids and Related Substances" is very short (9 pages) and does not do justice to the very extensive and thorough work of Ryhage and Stenhagen. This is all the more regrettable, because this first-class work, though published in English, has appeared almost exclusively in *Arkiv Kemi* and thus has not received the wide readership among organic chemists that it deserves. Furthermore, the Swedish group has been one of the principal ones responsible for the current interest in mass spectrometry on the part of natural product chemists.

In striking contrast to the short Chapter 6 are the 36 pages of Chapter 7 dedicated to amino acids and peptides. This is an area where Biemann and collaborators have made significant contributions, although it is regretful that the parallel investigations of Ryhage, Stenhagen and their colleagues (*Arkiv Kemi*, **19**, 405, 417 (1962)) could not be mentioned. The mass spectral fragmentation behavior of amino acid derivatives (notably ethyl esters) is discussed nicely in great detail and its application to the structure elucidation of new amino acids is illustrated by several examples. Though published some three years ago in a joint paper by Biemann, Lederer and their colleagues, the detailed analysis of the structure determination of lysopine is very welcome as it is of considerable pedagogic significance. The final portion of this chapter deals with potential applications of mass spectrometry to sequence analysis of small peptides, especially after lithium aluminum hydride reduction, and appropriate illustrations from the author's own papers are again cited.

Alkaloids, covered in Chapter 8, represent an area where Biemann has made very important contributions, and the utility of mass spectrometry in this field is best testified by the observation that this chapter is already the most out-of-date one in the entire book. This excellent discussion starts with the less important problem of identification of drastic degradation products (*e.g.*, pyridines from zinc dust distillation) and then proceeds to the very significant use of the mass spectrometric shift technique in the structural recognition of a given alkaloid by mass spectral comparison with a relative, differing only in the substitution of the aromatic portion of the molecule. Principal attention in this section, as well as in the following one dealing with the interpretation of the mass spectra of such alkaloids, is paid to the *Iboga* bases and to relatives of aspidospermine—two groups on which Biemann and Spittler have published notable papers.

A few of the mechanistic conclusions may be attributed to the above-mentioned overenthusiasm of attempting to rationalize too many features of the spectra. Thus, the statement on p. 316 that the loss of the angular ethyl group in aspidospermine "is not favored as it (the ion) would be located on a bridge head" is meaningless, since there is nothing wrong with this particular type of "bridge head" ion, which is simply a standard tertiary cation. This is clearly demonstrated on p. 325 in the mass spectrum of a close relative, where such a loss of ethyl is very pronounced. The explanation is simply that fragmentation of ring C in aspidospermine with expulsion of ethylene results in a greater energy gain than loss of the angular ethyl group. Similarly, on p. 330 a cleavage (B in structure (31)) is proposed for a peak, which does not exist in the mass spectrum (Fig. 8-13) of the alkaloid, although its presence is required by the postulated mechanism. However, these are minor faults in a chapter which describes well the status (until the beginning of 1962) of a field which has been progressing extremely rapidly since that time and where at least two dozen pertinent articles have appeared in the interval.

Chapter 9 ("Steroids and Related Substances") is perhaps the least satisfactory and least complete chapter in the book, probably reflecting the author's own interest in other areas. A number of the recent pertinent papers from the literature were not or could not be cited and several fragmentation mechanisms are based on very tenuous grounds. Indeed some of them (e.g., discussion of 11-keto steroids on p. 347) have been disproved in the reviewer's laboratory by recent studies with deuterium-labeled steroids.

The last two chapters, entitled "Miscellaneous Classes" (7 pages) and "Applications to Synthetic Problems" (4 pages) represent only very sketchy outlines, with the exception of a useful discussion of recent work on nucleosides performed in the author's own laboratory.

The production and printing of the book follow the usual high standard established by the McGraw-Hill Book Company. The proofreading, on the other hand, has been carried out in a somewhat sloppy manner and the book contains a substantial number of trivial errors. Some of them are consistent misspellings throughout the book, which could have been caught by the editorial staff (furan, pyran, chlorohydrin, etc., spelled with a terminal *e*, deuterio spelled without the *i*—all of them insignificant but annoying because they appear so often) and so could the printer's own notation on p. 118. There are minor errors in the figures on pp. 47 (C_6H_5N), 272 (N-formylisoleucine) and 332 (M-43 misplaced), methyl groups missing in squalene (p. 247), extra carbomethoxy groups in structure (22) on p. 324, equations (3-36) and (3-37) reversed on p. 108 and bond *a* (mentioned in text) not marked in formula, as well as trivial spelling errors on pp. 108, 218, 235, 241, 275, etc. The peaks at m/e 176 and 178 mentioned on the top of p. 101 actually refer to m/e 146 and 148 in Fig. 3-16. These are only beauty marks, but they are unnecessary.

In conclusion, where does this leave the potential reader for whom this book is largely intended—the organic chemist with a minimum or no knowledge of the role of mass spectrometry in organic chemistry? I would recommend that such an individual start out with McLafferty's chapter (*loc. cit.*), which covers the organic chemist's field clearly and succinctly. Following this introduction, he should delve into Biemann's book, possibly in conjunction with the more extensive text of Beynon (*loc. cit.*), which can be employed for supplementary reading on those sections (e.g., instrumentation, ionization potentials, etc.), where no detailed coverage was intended in the present book.

Actually, my view is that the ideal book on mass spectrometric applications in organic chemistry cannot be written for at least another two to three years, because there still exist too many gaps in too many areas, which however are being clarified at an ever accelerating pace. In another three years, the chapter on synthetic applications, instead of its present 4 pages, will be substantial; the mechanistic sections will be replete with well-documented cases based on labeled substrates; and, finally, many additional organic structures will have been subjected to the type of semiempirical approach that has created the presently existing body of knowledge. It is questionable whether at that time a single author will have the stamina and background to prepare such a book for the organic chemist. However, if single authorship is possible, Biemann may well be the author *par excellence* for that future and necessary monograph.

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Theories of Electrons in Molecules. By WILLIAM T. SIMPSON, Professor of Chemistry, University of Washington. Prentice-Hall, Inc., Englewood Cliffs, New Jersey. 1962. vii + 183 pp. 16 × 23.5 cm. Price, \$9.00.

Advanced students of quantum chemistry will welcome Professor Simpson's book and will benefit from it. The expressed purpose of the book is to "fill in the gap between what is found in quantum chemistry books and what is found in the journals"; however its importance probably lies more in the fresh ideas and insights that it will bring to those who are already cognizant of the literature. Indeed, one cannot expect a book of this length to fill more than a few selected gaps. Also, it should be pointed out that the level of presentation is not noticeably below that of most chemical papers, and the reader is expected to be familiar with transformation theory and group theory.

The book is an integrated collection of special topics related to the author's research interest rather than an introduction to or survey of molecular quantum mechanics. Emphasis is placed on mathematical rigor; however the physical problems are always kept close at hand. Professor Simpson proceeds immediately into the calculation of matrix elements in the first pages, and a rapid pace consisting of concise statements, equations, and little wasted space is maintained throughout. Chapter 1 is mostly concerned with the quantum theory of atoms while Chapters 2 and 3, which are somewhat longer, are devoted to the molecular

orbital method and the valence bond method, respectively. The treatment of the valence bond method is especially good. The independent systems approach is briefly discussed in Chapter 4 where, unfortunately, the novel molecules in molecules method is confined to one short section. In addition, a large amount of information is compressed into the twelve sections which make up the appendices and the treatment again is concise. The topics covered in these sections range from the Born-Oppenheimer approximation to time dependent polarizability.

Usually the presentation is clear, though at times overly abbreviated, and the derivations and illustrations are often original. The examples, which are worked out in many sections, are exceedingly useful. Although a few typographical errors are present, they do not detract from the usefulness of the book.

I strongly recommend this book to advanced students of quantum chemistry and to spectroscopists with sufficient background. The non-experts will find that it is not easy reading and that step-by-step verification with pencil and paper will often be required; however their labors will be well rewarded.

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Inorganic Polymers. Edited by F. G. A. STONE, Department of Chemistry, Queen Mary College, University of London, London, England, and W. A. G. GRAHAM, Arthur D. Little, Inc., Cambridge, Massachusetts. Academic Press Inc., 111 Fifth Avenue, New York 3, N. Y. 1962. xi + 631 pp. 16 × 23.5 cm. Price, \$19.50.

This book was written to provide chemists with a reliable review of current research in several areas of inorganic polymer chemistry. It consists of nine chapters contributed by leading industrial and academic scientists. As pointed out in the Preface the state of knowledge of inorganic polymers is not as well developed as it is for organic polymers. Much of the effort in this field at the present time is devoted to the study of the basic chemistry of model systems and the book reflects this by frequently discussing simple, even non-polymeric, inorganic systems.

In Chapter 1 (A. V. Tobolsky) pertinent polymer properties are defined and briefly reviewed. Obviously the objective of this chapter was to acquaint workers in the inorganic polymer field with some of the fundamentals on which the organic polymer chemist bases polymer evaluation. Many readers will be prompted to dig deeper.

The second chapter (J. R. Van Wazer and C. F. Callis) on "Phosphorus-Based Macromolecules" draws very heavily on the book "Phosphorus and Its Compounds, Vol. I" by Van Wazer. Thus, of the thirty-four figures in this chapter twenty-four are credited to this source. The discussion of the polymeric phosphonitrilic chlorides—essentially word for word the discussion in the above treatise (published in May, 1958)—does not take into account the well over one hundred papers published since 1958. As a consequence, the views presented on this unique, completely inorganic system exhibiting typical polymer properties are not up to date. The discussion on the polymerization of the lower phosphonitrilic chlorides, for example, is based entirely on the work by Patat, *et al.*, and ignores the results of later investigators such as Konecny and Douglas, Gimblett, etc. Becke-Goehring's work on linear phosphonitrilic chloride polymers endcapped by HCl—cited as a personal communication (ref. 6)—was published in 1959 (*Chem. Ber.*, 92, 1188 (1959)). This reviewer—who may be biased—would have preferred coverage of the phosphonitrilic polymers similar to Schmulbach's review in "Progress in Inorganic Chemistry" (Vol. 4).

Chapter 3 ("Sulfur Polymers," by M. Schmidt) is limited to compounds containing sulfur-sulfur bonds with the additional arbitrary restriction that at least one of the sulfur atoms must be bivalent. After a rather detailed up-to-date discussion of the various forms of elementary sulfur—which incidentally is also briefly covered in Chapter 2—the chapter mainly reviews the basic chemistry of polysulfides, polythionates and related systems.

Boron polymers are reviewed in Chapter 4 by A. L. McCloskey. The reader may stumble over an apparent contradiction: on p. 161 borazole is said to be thermally stable at 500° whereas on page 163 the statement is made that "borazole deposits solids on standing or on being heated, and gives off hydrogen under the same conditions." It should also be pointed out that references 54 and 63 report not only on the volatile products from the pyrolysis of borazole, but also on non-volatile residues of compositions $BNH_{0.8}$ and $BNH_{0.3}$, contrary to the statement on p. 163 "These residues have not since been investigated."

Silicones are the only inorganic polymers that have so far reached commercialization. There is, of course, much more information available on these polymers than on any other inorganic polymer system. They are admirably reviewed by A. J. Barry and H. N. Beck in Chapter 5 which almost could have