Microanalytic Methods

The Electron Microprobe (Proceedings of a symposium sponsored by the Electrochemical Society, Washington, D.C., October 1964. T. D. McKinley, K. F. J. Heinrich, D. B. Wittry, Eds. Wiley, New York, 1966. 1051 pp., illus. $27.50) presents some 43 papers ranging in scope from the basic physics of electron and photon interaction with solids to analysis of aerospace electronic components. Approximately half of the articles are devoted to applications of the microprobe. The importance of the microprobe in the analysis of semiconductor and electronic components is underscored. In the five papers on this topic, the authors have taken care to include the microprobe procedures in detail as well as the results. Many of the other applications presented deal with metallurgy. In 15 years, the microprobe has essentially revolutionized the venerable art of metallography; as ten papers on the topic indicate, virtually any microconstituent can now be qualitatively identified. The use of scanning displays, a television-type device in which the distribution of various constituents can be accurately mapped and recorded in a matter of minutes, is widely illustrated.

Although the microprobe has been applied in biology and medicine for nine years, only two papers in these fields are included. A possible reason is that analysis of the elements between boron and sodium—the most important range in biology—by means of their x-ray spectra was not feasible until recently. Six papers devoted to light-element analysis describe these difficulties and means for overcoming them. In fact, this group of papers is sufficiently detailed to serve as a basic text for those interested in analysis by means of low-energy x-rays.

New instrumentation is described in a special section. Three manufacturers of commercial microprobes introduced new instrumentation at the symposium. Now, two years later, this "new" material is of no great current interest, and its inclusion does not add to the scientific worth of the volume. Also in this section, preliminary studies with a combined electron microprobe-microscope are presented in which morphological, structural, or chemical information about small precipitates may be obtained at will.

Of primary interest are the 12 papers devoted to the topic of quantitative chemical microanalysis. Since Castaing stated in 1951 that quantitative analysis required only elemental standards, data-correction models and procedures have been the subject of controversy. Furthermore, required correction input parameters such as x-ray mass attenuation coefficients and fluorescent yield factors are often not well known. Perhaps the basic issue is joined in the presentations of this volume: use of full computer programs with electron trajectories calculated from basic theory or by Monte Carlo calculation, or use of shorter, so-called pencil-and-paper corrections based on semi-empirical derivations. This issue is still in doubt some two years after the symposium. However, the work described has served as a base for much further study of correction procedures. Especially valuable are the x-ray mass attenuation coefficients tabulated by Heinrich (p. 350 ff). At least one paper deals with the hitherto almost neglected role of the effect of the behavior of the detector system on quantitative analysis. Finally, there is appended a bibliography, compiled by K. J. F. Heinrich, which contains over 1000 references up to January 1965. To the working microprober, such a bibliography is indispensable.

One of the two major faults of the volume is the long period between the symposium and publication of the proceedings. In a rapidly changing field, the significance of several of the papers has diminished. The other fault is that no portion of the discussion which followed each paper is reproduced. However, in a field in which the contributions are widely scattered, a collection of 43 papers is a most welcome addition.

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Instrumental Analysis in Chemistry

In its sixth edition, *Standard Methods of Chemical Analysis* has been expanded by the addition of an entirely new third volume, **Instrumental Methods** (Frank J. Fletcher, Ed. Van Nostrand, Princeton, N.J., 1966. Part A, 992 pp., illus.; part B, 1056 pp., illus. $25 each). The volume includes 64 chapters written by 84 contributors and is bound in two parts. In part A, each of the first 41 chapters is devoted to a single type of instrumental method. The remaining 5 chapters, and the 18 in part B, are concerned with applications of the various instrumental methods to a particular industry or field of analysis.

The first part of volume 3 covers a remarkably broad spectrum of instrumental methods, including not only the commonly treated techniques of spectroscopy, electroanalytical chemistry, chromatography, and thermometric analysis, but also several topics not often encountered in the ordinary book concerned with instrumental methods; among these topics are particle size analysis, sedimentation analysis, electrophoresis, critical solution temperature, electron-spin resonance, magnetic susceptibility, and electron and chemical microscopy. Despite the fact that 40 or more authors contributed these chapters, I found a reasonable degree of uniformity among them. In each instance, there is a brief treatment of principles, followed by a discussion of instrumentation and commercially available equipment. So far as possible, detailed directions for performing an analysis by the technique are given; finally, a tabulation of applications, with references to the second part of volume 3, is included. In general, the emphasis in these chapters is on the practical; in most cases, the authors have attempted to give an honest evaluation of the accuracy of the method and not only its strengths but its limitations as well. With this emphasis on practicality and with chapters limited to 15 to 30 pages, it is clear that the treatment of principles must be abbreviated and largely qualitative. These theoretical sections are, however, liberally annotated with references to recent review articles and monographs directing the reader to the best sources of more detailed information about the subject matter.

The chapters in the first part of volume 3 could conceivably serve as a text for a survey course in instrumental analysis. They should also prove useful as a starting point for the chemist interested in developing a working knowledge of one or more of the techniques discussed.

The second part is concerned with details of applications of various instrumental methods to industrial products, to medicine, and to pollutants. The topics are largely the same as those treated in the second part of volume 2, with the emphasis in this case on instrumental procedures. The variation in quality and depth of treatment is much more pronounced in this second section of volume 3 than in the first. For example, in some chapters sufficiently detailed instructions are found for the performance of a given analysis so that recourse to the original literature may be unnecessary. Other chapters are quite general in their treatment and largely serve as a catalogue of literature references.

In my opinion, the attempt to separate the application sections in this edition into two parts based on instrumental and noninstrumental procedures is regrettable. The artificiality of this division becomes apparent in a perusal of the two sections. In volume 2 (noninstrumental), for example, one finds procedures based on potentiometric or spectrophotometric measurements. In volume 3, frequent references must be made to details found in volume 2. If indeed a separation must be made (which I doubt), might not this separation be better based on "standard" and "nonstandard" methods? The so-called instrumental methods then fall primarily though not exclusively in the latter category; however, the distribution will undoubtedly change with time. The sixth edition of *Standard Methods* is a useful reference work and belongs in technical libraries.

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Stroboscopy

The stroboscopic, or pulsed-image, principle has been exploited, if not understood, for hundreds of years. A variety of mechanical devices have utilized this optical illusion to titillate the human observer. With the advent of the practical electronic flashtube some 35 years ago, stroboscopy entered a new era of practical application, including speed measurements, motion studies, photography, and stimulation of photosensitive materials. Pulsed light is a versatile tool of enormous potential value in a broad range of fields, yet few books have been written on the subject. Jerzy Rutkowski's **Stroboscopes for Industry and Research** [translated from the Polish *Stroboskopy* (Warsaw 1961) by E. Lepa. Pergamon, New York, 1966. 278 pp., illus. $12.50] brings together for the serious researcher a wealth of material on the theory of stroboscopy and the construction of stroboscopic instruments.

The presentation assumes a grasp of undergraduate mathematics, and the material is treated from a theoretical rather than a practical standpoint. The book will serve admirably as a text because of its in-depth treatment. For example, the discussion of the photometric properties and perception of the pulse image will be welcomed by disciplines ranging from engineering to physiology.

The still-useful mechanical stroboscope is described in detail, and there is exhaustive coverage of the electrical circuits and components employed in flash stroboscopes. Considerable attention is paid to circuit functions, which are explained in terms of their complex interrelationships in practical stroboscopes.

The important subject of synchronization is well covered, as are the characteristics of various accessories. But the material on applications is weak, and the tabulations of available equipment with their abbreviated characteristics around 1960 are already dated. It seems unlikely that the book will receive the universal acceptance suggested by inclusion of "industry" in the title.

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Chemistry of a Functional Group

The Chemistry of the Carbonyl Group [Interscience (Wiley), New York, 1966. 1039 pp., illus. $32.50] is the second in a series of volumes produced under the editorship of Saul Patai which attempt to deal exhaustively with the chemistry of functional groups. Its predecessor, which dealt with the alkenes, was enthusiastically received in these pages a year ago; I cannot accord a similar welcome to the present volume. This volume seems to be the victim of two current conceptions regarding the production of scientific books: that of publishers which holds that libraries will acquire willy-nilly anything presented to them regardless of price or quality, and that of editors which maintains that their task is complete when signed contracts have been secured for a reasonable number of chapters. All in all, the book is not really bad; neither is it nearly as good as it might have been.

This treatise, which attempts "to encompass all facets of a functional group and to give up-to-date descriptions of the nature of the carbonyl group, of the main pathways leading to its formation, and of its main modes of reaction," consists of 17 loosely coordinated chapters contributed by a total of 26 authors. Admittedly, organizing the efforts of so many to produce a coherent whole is an ambitious goal; it is one which is not realized in the present case. The scope of the volume is so broad that, despite its length, important topics are dealt with superficially, redundantly, or not at all. For example, a topic so basic to carbonyl chemistry as the reaction of these substrates with amines nowhere receives a thorough discussion. The forward reaction is discussed in one chapter, the microscopic reverse in quite another, and the analytical uses of these reactions in yet another. Of course, the entire subject is to be completely rehashed in an upcoming volume, so perhaps one is expected to forgive the present inadequacies. The closely related reactions involving the addition of oxygen nucleophilic reagents to carbonyl compounds seem to have escaped attention altogether. Similarly, the reasonable entry "eneamines" appears nowhere in the index; this is not the fault of the index.

One wonders whether chapters such as that dealing with general and theoretical aspects of the carbonyl group really belong in a book of this type. The chapter is competently done, but those interested in quantum mechanical treatment of carbonyl compounds are not likely to resort to a volume aimed principally at synthetic organic chemists. A similar question may be raised concerning a chapter on chemical and physical methods of analysis. The space occupied by such material might better have been employed to provide additional depth to topics central to the main theme of the entire work. Lack of really adequate coverage is ac-