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.