

Book Review

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Comprehensive Analytical Chemistry. Volume IIB. Physical Separation Methods, edited by C. L. WILSON AND D. W. WILSON, Elsevier, Amsterdam, 1968, 445 + XV pp., price Dfl. 70.

This latest volume in this series is devoted entirely to methods for the physical separation of mixtures, and contains chapters on liquid chromatography in columns, gas chromatography, ion exchangers and distillation; paper chromatography and thin-layer chromatography have had to be left to a later volume.

Over half of the chapter on *Liquid Chromatography* (J. F. K. HUBER) is devoted to theory and the remainder relates the theory with the practical side of operating liquid chromatographs, e.g. on the choice of adsorbent and solvent/solvent mixtures, flow rate, temperature, sample size and detector types and performance. Interest in liquid chromatography has been revived recently by the development of several new and very sensitive detectors, and this chapter is therefore a timely one: it is reasonably up-to-date (but briefly) on detectors, although Table I, 7 is little help in comparing the sensitivities of the new detectors because there is no uniformity as yet in the literature in expressing sensitivity data. There are no examples of the applications of liquid chromatography.

Gas Chromatography (E. R. ADLARD, R. STOCK AND B. T. WHITHAM) is covered in Chapter II in 150 pages, and within this severe limitation it is a reasonably comprehensive account; the theoretical section is kept short, yet it will be sufficient for most practical people. The apparatus section and the subsequent sections on solid supports, stationary phases, adsorbents (for gas-solid chromatography) and the preparation of columns are full of useful background information. The chapter then gives details of the retention volume method for identification (but does not give a strong warning of its limitations) and some brief remarks on fraction collecting for spectroscopic identification. The quantitative analysis section is brief and omits two rather useful methods of reasonable precision, namely (a) "bracketing" between two known mixtures, and (b) adding a known amount of the impurity being determined (the correct calculation for which is given by J. NOVÁK AND J. JANÁK, *J. Chromatog.*, 28 (1967) 392); this section could usefully give one or two examples showing the precision attained.

Further sub-sections on the operating procedures used for gas samples, liquid samples, solid samples and polymers usefully summarise most of the important factors. The final section on applications gives brief summaries of the literature under appropriate headings.

The chapter as a whole has rather too much detail about some less important matters (e.g. three drawings of 11-year old katharometer apparatus, Janák's method) and some repetition (e.g. in pp. 143-165); several important subjects are omitted,

such as the thermal flame ionisation detector for phosphorus compounds, support-coated open tubular columns, the Curie point pyrolyser, and the merits of on-column or preheater injection. More information should have been given on fraction collecting for spectroscopic identification, automatic preparative chromatographs and on combined gas chromatograph-mass spectrometer apparatus.

Despite the above criticisms, this chapter is a clear account of the principles and practice of gas chromatography which will undoubtedly be useful to those learning the technique or using it in their everyday work.

The chapter on *Ion Exchangers* (F. C. SAVILLE) discusses the mechanism of ion exchange, describes the experimental techniques and gives a substantial section on applications, with alphabetically-arranged subheadings. Appendix 1 gives a 20-page Table of data on commercially-available exchangers, and Appendix 2 gives detailed instructions for testing them. Both the Bibliography (Appendix 3, 307 refs.) and the References (225) are arranged with the same subheadings in alphabetical order. The subject is very clearly explained and much valuable information is given. Unfortunately, the applications section is not up-to-date, as for example in the section on amino acids, the latest reference being 1956.

After giving a 20-page section on theoretical background, the chapter on *Distillation* (G. A. DUMMETT, N. A. H. HOLT AND M. G. ROYSTON) gives a wealth of details on the experimental techniques; the first part, on simple distillation, refers to the methods of the STPTC, NBA, IP, and ASTM and describes the scope and limitations of distillation range tests; in the second part, on analytical fractional distillation, the types of fractionating columns and the ancillary equipment, are described at length; this is followed by sections on vacuum distillation, low temperature distillation, steam distillation and molecular distillation. Having reviewed the available apparatus, the authors then give a useful section on the selection of a column and on the techniques for charging and operating the still.

The book is written by several authors, and this has resulted in some repetition and lack of uniformity. In the reviewer's opinion, the book would have been better with an Introductory Chapter on general principles of chromatography, dealing with isotherms and partition, elution and displacement development, and theoretical plates and resolution. This would have avoided, for example, calculation of the number of theoretical plates in 3 different ways (*i.e.* using the peak width at half the peak height (p.27), the base width (p.72) and, on p.247, the width at 0.368 of peak height); it would also have eliminated the bulk of the repetition (of which Figs. II, 12 and III, 7 are examples) and could have led to more uniformity in the use of symbols.

The book is well produced and bound, with an adequate index and only a few typographical errors.

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