NEW BOOKS

edited by F. W. Quackenbush

LIPID CHROMATOGRAPHIC ANALYSIS, Vol. 1, edited by Guido V. Marinetti (Marcel Dekker, Inc., New York, 1967, 560 p, $23.50).

This book is the most up-to-date compendium of its kind with the following 14 chapters: Paper Chromatography of Phosphatides and Glycolipids on Silicic-Acid-Impregnated Filter Paper, by Morris Kates; Thin-Layer Chromatography of Phosphatides and Glycolipids, by O. Renkonen and P. Varo; Column Chromatographic and Associated Procedures for Separation and Determination of Phosphatides and Glycolipids, by George Rouser, Gene Kritchevsky, and Akira Yamamoto; Analysis of Phosphatides and Glycolipids by Chromatography of Their Partial Hydrolysis or Alcoholysis Products, by R. M. C. Dawson; Thin-Layer Chromatography of Neutral Glycerides and Fatty Acids, by V. Mahadevan; Column Chromatography of Neutral Glycerides and Fatty Acids, by K. K. Carroll and B. Serdarevich; Gas Chromatography of Neutral Glycerides, by A. Kuksis; Isolation and Gas-Liquid Chromatography of Alkoxy Lipids, by Helmut K. Mangold and Wolfgang J. Baumann; Gas-Liquid Chromatography of Fatty Acids and Derivatives, by Robert A. Stein, Vida Slawson, and James F. Mead; Gas Chromatography of the Long-Chain Aldehydes, by G. M. Gray; Thin-Layer Chromatography and Gas Chromatography of Sphingosine and Related Compounds, by Benjamin Weiss; Gas Chromatography of Inositol and Glycerol, by Richard N. Roberts; Gas Chromatographic Estimation of Carbohydrates in Glycolipids, by Charles C. Sweeley and Dennis E. Vance; Chromatographic Analysis of Nitrogen Bases Derived from Lipids, by John M. McKibbin.

Each chapter contains an outline and table of contents. Some bibliographies are conveniently divided into specific references and general review articles. Both author and subject indices are given.

The chapter on silicic acid-impregnated paper chromatography does not indicate the true potential of the method because key literature references are omitted which critically evaluate the shortcomings of the procedures described for quantitative application and which present more reproducible systems and applications. Unfortunately, a number of the one-dimensional chromatograms used for illustration show typically incomplete resolution.

Thin-layer chromatography of phosphatides and glycolipids is presented in an excellent review which devotes appropriate attention to the separation of molecular species within a given lipid class. Excessive attention is directed to lipid extraction and purification methods which are unsatisfactory for quantitative purposes.

The chapter on column chromatography of phosphatides and glycolipids is almost in itself worth the price of the book. Completely detailed procedures and the necessary precautions are presented for obtaining precise, reproducible, quantitative analyses of complex mixtures of polar lipids. The authors show how different types of columns may be combined to accomplish specifically desired separations and how these may be combined with thin-layer chromatography and gas-chromatography for complete quantitative analyses. Modifications necessary for application to specific tissues are also presented. Details for the general use of reproducibly packed and not easily overloaded TEAE-cellulose columns recorded in this chapter are not published elsewhere in the literature.

The chapters on partial hydrolysis and alcoholysis products of phospholipids and the chromatographic analysis of nitrogen bases derived from lipids provide methods useful for confirming the identity of phospholipids isolated by other methods and possibly in establishing the identity of new lipids. Some of these methods offer an approach to the problem of estimating the amounts of plasmalogen and alkoxy-ether forms of an isolated phospholipid class.

The chapters on column chromatography of neutral glycerides and fatty acids and on thin-layer chromatography of neutral glycerides comprehensively review the literature and critically evaluate the degree of resolution possible. Modifications of thin-layer chromatography for preparative and quantitative analytical purposes are presented. Detailed procedures for detection, isolation, and fractionation of alkoxy lipids and the preparation of suitable alkyl glycerol ether derivatives for analysis by gas-chromatography are separately described in another chapter, although the quantitative nature of this approach is not appraised.

A wealth of experience is presented in the chapter on gas chromatography of neutral glycerides with a detailed consideration of the theory of temperature programmed gas-liquid chromatography and its application to glycerides. Parameters are comprehensively review for column selection, preparation, operation, and evaluation for both quantitative analytical and preparative gas-liquid chromatography and the preparation of derivatives. The potential of the method is illustrated, particularly when combined with other methods, by a number of applications to analysis of natural mixtures, structure determinations, direct analyses of milk, lymph, plasma, and elucidation of phosphatide structure.

The chapters on gas-liquid chromatography of fatty acids and derivatives and long-chain aldehydes are comprehensive reviews of the effects of substituent groups on the retention behavior of fatty acids, their isomers and derivatives, methods of preparation of methyl esters, collection of methyl esters, problems in quantitation of methyl esters, methods for isolation of aldehydes, synthesis of long-chain aldehyde standards, derivative preparation, chromatographic conditions, and some applications to biochemical problems involving long-chain aldehydes.

Thin-layer and gas chromatography of sphingosine and related bases and derived compounds is concisely and thoroughly reviewed with essential experimental details. A number of biological applications is presented although the quantitative nature of the methods is not appraised.

Gas chromatography of inositol and glycerol is presented in an excellent manner with proper attention devoted to hydrolytic release, derivative preparation, methodological details, and a critical evaluation of the precautions essential to assure quantitative results.

The chapter on gas chromatographic estimation of carbohydrates in glycolipids is addressed to the problems of quantitative liberation of carbohydrates from glycolipids, recognition of individual carbohydrates from a given glycolipid, and accurate determination of the relative proportion of each of the individual carbohydrates in a mixture of complex glycolipids. The shortcomings and limitations presented suggest that these methods require further development for quantitative reliability.

This volume is indispensable to biochemists, clinical and analytical chemists associated with research in lipids and will be useful to processing, industrial, food, and manufacturing chemists concerned with quality control. The prospective second volume is eagerly anticipated if it is of the quality of the first.

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The 1968 Chevreul Award

Every year, since 1963, the French Association of Fat and Oil Technicians (Groupement Technique des Corps Gras) awards the Eugène Chevreul medal to French or foreign leaders in the field of lipid research.

On May 28, 1968, the medal was awarded to MARIE-THÉRÈSE FRANCOIS, Professor at the University of Nancy, France, and to JAN BOLDINGH, member of the Royal Netherlands Academy of Sciences, Professor at the University of Nimwegen, and Manager of the Unilever Research Laboratory, The Netherlands, for their valuable contribution to the development of knowledge in the field of fats and oils.

Since 1963 the Chevreul medal has been awarded to G. Jacini (Italy), G. Champetier (France), M. Loncin (Belgium), T. P. Hilditch (Great Britain), P. Desnuelle (France), A. R. Baldwin (U.S.A.), J. Martinez Moreno (Spain), H. Niewiadomski (Poland), C. Paquot (France).

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INFRARED BAND HANDBOOK, Supplements 3 & 4, by Herman A. Szymanski (Plenum Press, Data Division, New York, N.Y., 1966, 261 + xiv pp., $15.00).

As with the original volume and Supplements 1 & 2 (See Reviews JAOCS *41*, 32, May 1964, and JAOCS *43*, 73A, February 1966), this handbook consists entirely of tables, 20 to a page. The main portion of the book (245 pages) is, like the earlier volumes, a uniform presentation of infrared absorption band data, arranged in order of decreasing wavenumber. In the center of each table, the molecular structure of a specific compound is reproduced. In the upper left-hand corner, the frequency in cm^{-1} of the specific absorption band is given followed by a letter indicating the band intensity as determined from reference to an intensity code given in the introduction. A second entry in the upper left-hand corner of each table indicates, by means of a single letter (again interpreted with the aid of a physical state code in the introduction), the physical state in which the spectrum was measured or the solvent used for the sample. This is followed by an indication in brackets of the concentration and cell thickness. The "Explanation" in the introduction indicates that this latter item is given "where pertinent," but it is, apparently, not often considered "pertinent" as cell thickness is very rarely indicated. A third line in the upper left-hand corner is reserved for an indication of the dispersive element used, if other than an NaCl prism. In the lower right-hand corner of each table, a reference is given to the original source of the data reproduced. A glance at the reference given at the end of the tables (page 247) reveals that all data have been obtained from four American and two German journals, during the three years 1963 to 1965. The lower left-hand corner of each table is reserved for an indication of the structural group to which the vibration has been assigned. However, these data, a glance over several pages of the text reveals, are available in less than one-half of the tables. No infrared spectra are reproduced throughout the volume.

Readers familiar with the original volume, who subsequently learned that Supplements 1 and 2 did not increase the probably insufficient number of bands listed in the original volume (approximately 8,500 in the rock salt region from 3610 to 621 cm^{-1}), but rather extended this range into the KBr or far infrared region from 600–200 cm^{-1}, will be interested to learn that Supplements 3 and 4 increase the number of bands by about 5,000 in both regions, covering bands from 4,200 cm^{-1} to 29 cm^{-1}. Only 65 bands are listed below 200 cm^{-1} (Supplements 1 & 2 listed some three or four dozen below 200 cm^{-1} down to 41 cm^{-1}).

The analytical spectroscopist can use infrared absorption spectra in one of two ways to identify an unknown compound, probably the principal use to which tabulations in this Handbook and its Supplements would be put. First, he can attempt identification by group frequency correlations of specific absorption bands with structural groups, usually referred to as the Julius' group frequency technique. Or he can attempt to find an exact match of the spectrum of his unknown with that of the spectra of a number of known molecules, the so-called Coblentz "fingerprint" matching technique. As no spectra are reproduced, only the first of these two methods is aided by data from these Handbooks. For this reason, it appears unfortunate that in a majority of the tables, there is no indication given as to the structural group giving rise to the specific band.

The original Infrared Band Handbook, published in 1963, listed bands in the rock salt region for about 1,000 compounds or only 2.5% of the 40,000 then available in the American Society for Testing and Materials' referenced spectra collection. The present Supplements 3 and 4 add only about 400 additional compounds (Supplements 1 and 2 merely increased the frequency range to include the far infrared). Thus, the Infrared Band Handbook and its four Supplements, include only 1,400 bands, while the ASTM referenced compounds have increased to over 100,000. Thus, despite what appears (to this reviewer at least) as almost Herculeon efforts of Dr. Szymanski, the percentage of ASTM referenced compounds, his Infrared Band Handbook represents has fallen from a very low 2.5% to an almost vanishing 1.4%. These figures raise some doubts as to the future of these Handbooks. Can the amount of data ever be increased so as to represent a sufficient number of compounds to afford qualitative identification? Even if the mass of data could be so increased, would it not, long before it reached a worthwhile percentage of the compounds whose spectra have been measured, be so cumbersome and massive as to defeat its purpose for use as an aid to qualitative identification? And finally, with systems which convert the data to digitalized computer language on magnetic tapes or disks and permit the complete search of 100,000 compounds for several bands matches within 1, 2, or 4 seconds, can continued efforts for compilation of data for hand search through multivolume handbooks be justified?

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RAMAN SPECTROSCOPY, THEORY AND PRACTICE, Herman A. Szymanski, Editor (Plenum Press, New York, N.Y., 255 + IX pages, $12.50).

It is now just about two generations since Sir Chandrasekhara Raman discovered the phenomenon which bears his name. Almost continually during this period serious students have attempted to show how the effect was a natural complement to infrared (vibrational) absorption

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spectra. Raman spectra, it has been demonstrated, would provide the analytical chemist with the vibrational data from symmetrical molecules, which could not be obtained by means of infrared absorption requiring a change in dipole moment. For an equal period, analytical chemists have continued to ignore Raman spectroscopy, as any perusal of the literature verifies. Meanwhile, the literature of infrared absorption spectroscopy has grown to the point where bibliographies of its use, even when limited stringently to specific areas has become almost unwieldly.

Early research workers have probably no particular difficulty understanding the reluctance of analytical chemists to adopt Raman spectroscopy. The long struggles to obtain Raman shifts on photographic plates were more conducive to attempts to develop other methods of analysis. Nor did the introduction of direct reading instruments aid particularly in this deficiency. What Raman spectroscopy needed to become a satisfactory analytical tool was a new type of source which would be at the same time very intense and highly monochromatic. Then came the laser beam, whose major properties could be described almost exactly by the needs of a Raman source. As a result, forty years after its discovery, Raman spectroscopy appears to be ready to assume its promise as a useful tool of the analytical chemist, a fact that can be verified by a perusal of more recent programs concerned with analytical spectroscopy.

Dr. Szymanski appears to have come forward with a text which is very timely. The renaissance of Raman spectroscopy from its Dark Ages certainly has created the need for a text on the subject. While "Raman Spectroscopy, Theory and Practice" may lack some of the features ideally suited to accomplish their needs, the vast army of analytical chemists now ready to take this particular scattering effect into their laboratories, where spectroscopic instrumentation has become a familiar sight, will welcome the appearance of the first new book on Raman Spectroscopy for several years.

The first three chapters set a fast pace to achieve the interest of the analytical chemist. The initial Chapter entitled "General Introduction," by L. A. Woodward, is a very readable discussion of the Raman effect, its experimental measurement and applications in chemical analysis. An introduction to the mechanical theory of light scattering, and the theories of the polarizability of Raman scattering, are included with, especially a section devoted to the relation of bond polarizability to bond character. John R. Ferraro gives, in Chapter 2, "Advances in Raman Instrumentation and Sampling Techniques," an excellent resumè of the development of direct-reading instruments and the struggle to find intense monochromatic light sources, leading to the development of the laser spectrometers. This is followed by an excellent section on sampling techniques, the material that is needed by the new generation of Raman Spectroscopists. J. A. Koningstein, in Chapter 3, has the interesting assignment of describing the Laser Raman spectrometer, its principles, advantages, and some experiments performed with it.

With Chapter 4, the reader encounters a rather abrupt change of pace. Understanding of this chapter will require a solid background in molecular spectra and more than a general acquaintance with the phenomena of the scattering of radiation, as Ronald E. Hester discusses the theory of Raman intensities and the nature of the chemical bond as obtained from intensity and polarizability measurements.

There appears to be a curious inversion of the last four chapters, 4 through 7. Following a chapter on more or less detailed theory, Chapter 5, by G. J. Janz and S. C. Wait, Jr., is a practical discussion of the applications of Raman spectroscopy to ionic melts. This is followed by a lengthy, the longest chapter in the book, 56 pages, of "Observed Resonance Raman Spectra," by Josef Behringer. The level of this discussion is perhaps more advanced than Chapter 4. The book ends with Chapter 7, another discussion of the practical use of Raman spectra, this time to complex ions in solution.

The balance of material between Theory and Practice appears to be a bit tilted toward the former. The first three chapters, an introduction and the excellent descriptions of instrumentation, require exactly 100 pages. The remaining 150, not including five given over to a rather complete index, are divided, 94 to the two chapters on theory, leaving only 56 for all descirptions of practical applications. Probably many analytical chemists will be disappointed to find specific detailed applications limited to only two types of molecules, ionic melts and complex ions. Surely, one has to believe that if Raman spectroscopy is to take its promised place beside infrared absorption spectroscopy, these can be only two of the least important applications, as they concern the large numbers of analytical chemists.

Based on its overall merits, the book is to be recommended. It surely belongs in the hands of any analytical chemist about to supplement his spectroscopic equipment with a Raman spectrometer. It probably should be seriously considered by analytical spectroscopists contemplating such a move, or even by the analytical spectroscopist, who does not as yet foresee the immediate acquisition of a Raman Spectrometer, if only to enable him to be more acquainted with what is being done with yet another analytical spectroscopic tool. If the promise of Raman spectroscopy as an analytical tool is about to be fulfilled, this will not be the last text to appear, but the working analytical spectroscopist will be more than ready for what is to come for having spent some time with Dr. Szymanski's contribution.

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