

chromatography on a cellulose column using the solvent *n*-butyl alcohol-acetic acid-water (5:2:3) (isolated yield, 60%). This product (III), which was homogeneous by paper chromatography (R_f , Table I), was, as expected, resistant to the action of pancreatic ribonuclease and gave quantitatively uridylyl-(3'→5')-uridine⁴ after an ammoniacal treatment. The latter was degraded by the enzyme quantitatively to uridine-3' phosphate and uridine.

The condensation of III (pyridine salt, 0.030 mmole) with 2',5'-di-*O*-acetyluridine-3' phosphate³ (0.120 mmole) in dry pyridine in the presence of DCC followed by an ammoniacal treatment and separation by preparative paper chromatography gave uridylyl-(3'→5')-uridylyl-(3'→5')-uridine (IV; R = uracil) in 80% yield as based on the amount of III used. The product was degraded completely⁶ by pancreatic ribo-

(6) Enzymatic degradation of IV (R = uracil) was performed using 7 optical density units (260 $m\mu$) while that of IV (R = adenine) using 9.6 opti-

nuclease to give uridine-3' phosphate and uridine in the ratio 2:1. Analogous condensation of III with *N*,2',5'-triacetyladenosine-3' phosphate⁷ (3-fold excess) gave adenylyl-(3'→5')-uridylyl-(3'→5')-uridine in 75% yield as based on III. This product was degraded quantitatively⁶ to adenosine-3' phosphate, uridine-3' phosphate and uridine by the *Lactobacillus acidophilus* R26 phosphodiesterase.⁸ The R_f values of different compounds are given in Table I.

INSTITUTE FOR ENZYME RESEARCH
THE UNIVERSITY OF WISCONSIN
MADISON, WISCONSIN

Y. LAPIDOT
H. G. KHORANA

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cal density units. By application of the total product on paper chromatograms, degradation was found to be complete. At the levels tested, therefore, the degradations by the specific enzymes provide conclusive proof of the exclusiveness of C₃'-C₅' internucleotidic linkages in the products.

(7) Prepared by the method used for acetylation of uridine-3' phosphate (ref. 3) but using 3-5 days at room temperature for the *N*-acetylation.

(8) W. Fiers and H. G. Khorana, *J. Biol. Chem.*, in press.

BOOK REVIEWS

Gas-Liquid Chromatography. Theory and Practice. By STEPHEN DAL NOGARE, Senior Research Chemist, Plastics Department, E. I. du Pont de Nemours and Company, Inc., Wilmington, Delaware, and RICHARD S. JUVET, JR., Associate Professor of Analytical Chemistry, Department of Chemistry and Chemical Engineering, University of Illinois, Urbana, Illinois. Interscience Division, John Wiley and Sons, Inc., 440 Park Avenue South, New York 16, N. Y. 1962. xviii + 450 pp. 16 × 23.5 cm. Price, \$13.95.

Dr. Dal Nogare is one of the top experts in gas chromatography in the U.S.A.; his name is particularly well known from his work in the practical introduction of temperature programming and from his bi-annual reviews on gas chromatography in *Analytical Chemistry*. The present book, coauthored by Dr. Juvet, is the result of a long and careful study and is highly welcomed; it can be expected that it will soon become the standard textbook for the practical gas chromatographers.

The book is composed of eighteen chapters. The introduction summarizes the different chromatographic techniques, and the next chapter gives the basic theoretical and practical information necessary for understanding the gas chromatographic process. The detailed discussion of the distribution theory is the subject of a separate chapter; continuously, the theoretical basis of retention and resolution is given with special emphasis on the conclusions important in practice. The discussion of packed column performance is based on the extended van Deemter-Jones equation.

Following these basic chapters, the book discusses the liquid, solid and mobile phases and the two basic instrumental components: sample introduction systems and detectors. Successively, the methods of qualitative and quantitative analysis and some individual techniques (capillary columns, high temperature and trace analysis, temperature programming and preparative chromatography). Finally, two chapters deal with non-analytical and special applications. Detailed relative retention tables (reproduction of the work of Scholly and Brenner¹) and a list of U.S. commercial chromatographs and trade names close the book; a detailed and well prepared subject index helps when using the book as a reference book.

In my opinion, the book of Dal Nogare and Juvet is a very accurate summary of our present knowledge in gas chromatography, and its main strength is that the theoretical treatments are always closely related to practice. This and the many references after each chapter (totaling 883) make the book very valuable for every-day work.

I agree basically with the whole text of the book, and my remarks deal only with minor questions. The following detailed discussion of my remarks intends to serve as a contribution to future revisions of the text in consecutive editions.

(1) N. Brenner and P. R. Scholly, in "Gas Chromatography," ed. by H. J. Noebels, N. Brenner and R. F. Wall, Academic Press, Inc., New York, N. Y., 1961, pp. 263-309.

The main question with similar textbooks dealing with subjects under rapid development is how up-to-date the compilation can be. In this respect, the authors did a remarkable job, because although the basic manuscript was evidently ready in the first part of 1961, they did include many of the most important newer publications up to 1962. Naturally, however, this could not be done in every part of the book; or, if it was done, the authors had no time in melting the additions together with the main part of the book. Thus, in some places, one feels that a sentence or paragraph was inserted later in the manuscript without logical transition from the previous paragraph, or that it was not possible any more to rewrite parts of the manuscript. I had, for example, the latter feeling at the part discussing the response of the flame ionization detector (pp. 218-221) where the material of the 1961 Lansing, Mich., meeting could not be included any more. Since a certain delay in the subject of a manuscript is always inevitable, one has to ask the question whether the publication of such important books could not be speeded up. At the present rapid development, publications of books 1-1.5 years after compilation of the manuscript is inadequate, since in this way, parts of the manuscript are more than 2 years old when the book is published. For example, in gas chromatography, a 2-year delay means 20% of the lifetime of the whole technique.

The second general problem with such textbooks concerns the symbols used. Particularly in gas chromatography, a very wide variety of symbols can be found for the same expression, and the authors decision to try to apply the symbols recommended by the I.U.P.A.C. Committee² is certainly welcome. I found only a very few cases where the application of the symbols is not accurate, e.g., in the case of V_M , which means the corrected retention volume of the mobile phase in the book; although, according to the I.U.P.A.C. nomenclature, it is the *uncorrected* retention volume of a non-adsorbed sample.

It was a good idea to discuss at the beginning the principles of a general purpose laboratory apparatus; however, the given schematic (Fig. 2-5) is not typical, nor is its "commercial" version (Fig. 2-7) available. It is possible that it was built specially for the authors by the particular instrument manufacturer, but it is not available for others (it is not even included in the list of commercial instruments at the end of the book), nor is a general purpose instrument.

In my opinion, pyrolytic techniques are discussed in the wrong place (pp. 251-252). The pyrolysis-gas chromatographic technique is a special method similar to the microcatalytic-chromatographic technique (reaction kinetics studies), and if proper terms are used, the polymers which are pyrolyzed cannot be characterized as "the samples," for in this case, the mixture of the volatile pyrolysis products is the sample. It is unfortunate that later when discussing briefly the application of gas

(2) D. Ambrose (Chairman), A. T. James, A. I. M. Keulemans, E. Kováts, H. Röck, C. Rouit and F. H. Stross, *Pure & Applied Chem.*, **1**, 177 (1960).

chromatography for reaction kinetics studies (pp. 378-381), the two most important basic references^{3,4} are missing.

When discussing the flame ionization detector, the presentation of some basic plots (*e.g.*, response *vs.* H₂/O₂ ratio, etc.) is recommended. It is true that the authors refer to some publications where such plots can be found, but they belong to the basic discussion of this type of detector and the visualization of these correlations is a great help for the average reader.

I disagree with the present place of the chapter on "capillary" columns (pp. 267-284) (they should be discussed right after the performance of packed columns) and with the use of the term "capillary columns." These particular columns are not restricted at all to capillary diameters; on the other hand, their theoretical treatment (discussed in this chapter) is not valid for packed capillary columns, which are also described recently in the literature. Thus, the authors have to use once (p. 267) the absurd expression of "capillary capillary columns." The correct term is "open tubular columns" which can be prepared in a wide variety of diameters from capillary dimensions up. For sake of historical accuracy, I want to mention that in my opinion, Dr. Martin's "prediction" (p. 267) did not refer to *open tubular* columns.

I do not understand why Golay's expression⁵ for the possible minimum HETP of open tubular columns

$$\text{HETP}_{\text{min}} = r \sqrt{\frac{1 + 6k + 11k^2}{3(1 + k)^2}}$$

(where r is the column radius and k the partition (capacity) ratio) is not mentioned during the discussion of the theory of these columns. It describes excellently the influence of the open tube's radius and of the peak's relative position, and it is also very useful when studying actual column performances in comparison with the possible ideal values.

The chapter on the separation of high boiling compounds (pp. 285-299) is evidently an earlier part of the manuscript. Today, such a detailed chapter would not be necessary, since most of the new commercial chromatographs can be used up to 3-400°; thus, only the differences in the applicable liquid phases deserve a brief discussion. This could, however, be done as well during the general discussion of the liquid phases.

The chapter on trace analysis (pp. 300-318) is very useful, and particularly the discussion of the correlation of sensitivity and column resolution, since most people like to think *only* of the detector when dealing with trace analysis. The sensitivity of the ionization detectors (p. 307) would deserve a little more detailed discussion, and the possibilities of increasing the sensitivity of such GC systems by increasing the flow rate should be mentioned; the ionization detectors namely differ in this fact from the TC cells. The references on low temperature trapping (pp. 310-311) and on the use of precolumns (pp. 311-313) and the discussion of some techniques are somewhat mixed-up. One reference⁶ belongs to low temperature trapping and has nothing to do with the subtraction technique; on the other hand, the latter method⁷⁻¹⁰ was aimed rather for sample component identification than for application in trace analysis. Further, when quoting the work of Krejčí, *et al.* (p. 313), the explanation is incorrect because, argon and oxygen *can be* resolved on a Molecular Sieve 5A column if it is sufficiently long and is used at low temperatures.

As mentioned, these remarks do not intend to reduce the value of the book. I want to repeat my opinion that the present book is very valuable for the practice and is highly recommended to the practical gas chromatographers.

(3) R. J. Kokes, H. Tobin, Jr., and P. H. Emmett, *J. Am. Chem. Soc.*, **77**, 5860 (1955).

(4) W. K. Hall and P. H. Emmett, *ibid.*, **79**, 2091 (1957).

(5) M. J. E. Golay, in "Gas Chromatography 1958," ed. by D. H. Desty, Butterworths, London, 1958, pp. 36-55.

(6) N. Brenner and L. S. Ettre, *Anal. Chem.*, **31**, 1815 (1959).

(7) N. Brenner and V. J. Coates, *Nature*, **182**, 1401 (1958).

(8) B. T. Whitham, *ibid.*, **182**, 391 (1958).

(9) N. Brenner, E. Cieplinski, L. S. Ettre and V. J. Coates, *J. Chromatography*, **3**, 230 (1960).

(10) L. S. Ettre and N. Brenner, *ibid.*, **3**, 235 (1960).

GC INSTRUMENT APPLICATIONS
THE PERKIN-ELMER CORPORATION
NORWALK, CONNECTICUT

L. S. ETTRE

Gas Chromatography. By JOHN H. KNOX, Lecturer in Chemistry in the University of Edinburgh. John Wiley and Sons, Inc., 440 Park Avenue South, New York 16, N. Y. 1962. viii + 126 pp. 12.5 × 19 cm. Price, \$3.25.

The number of textbooks on gas chromatography is growing, but most of them are general textbooks. The present small booklet intends to serve as a *short compilation* of the subject.

The book is divided into seven chapters. After a brief introduction, the theory of gas chromatographic separation is discussed in a separate chapter followed by information on columns

and column packings, detectors, ancillary equipment and special gas chromatographic methods. The book is concluded by a short description of the most important GC books and a good subject index.

As mentioned in the preface of the book, its purpose is to give an adequate coverage to the new aspects (capillary columns and ionization detectors), and to present at the same time the basic principles of gas chromatography with sufficient details on apparatus construction in order to allow the reader the fabrication of his own instrument. In my opinion, the most objectionable part of the book is exactly this intention. I do not believe that the very brief descriptions on the construction of different instrumental parts are sufficient for somebody who actually wants to build his own instrument; at the same time, the philosophy that it is very easy to build a gas chromatograph results in some oversimplification. For example, when mentioning temperature programming (pp. 106-110), the author is forced to the very objectionable remark that precise control of the programming is really not required because otherwise he could not recommend simplified solutions for a home-made instrument. My feeling is that the usefulness of the book could be improved if more emphasis would be taken on the operation of the instruments and on the practical interpretation of the theories, and less on the possibilities of instrument building.

Besides this general problem, the book is a very useful brief discussion of the subject, and it can easily be read. In some details, however, I do not agree fully with the author's remarks.

It is not true that the katharometer detector was introduced in gas chromatography by Ray (p. 4). The first gas (solid) chromatographs consisting of a sampling device, a column and a katharometer detector were built by Cremer, Prior and Müller in the second part of the forties, and they even exhibited a primitive breadboard model at the 1952ACHEMA exhibition in Frankfurt.

It is not fully understood what the author means under the "same speed" of packed and open tubular ("capillary") columns (p. 7). If it means the column performance, I am in disagreement, because Golay proved that an open tubular column has fundamentally a better performance. I also have to repeat my remark made in the previous review on the name "capillary columns": This expression is incorrect and the term "open tubular columns" should rather be used.

The discussion of the support materials (pp. 45-47) is somewhat restricted; a very detailed method is given on how to silver plate or coat with polymer the Firebrick, but at the same time, the different kinds of diatomaceous earth-type supports (*e.g.*, the Chromosorb series) are not mentioned at all (except Celite) although they belong in a practical handbook. The very important method of silane-treatment (which is much more important than the silver plating) of the support is missing, although it should at least be mentioned.

The given temperature limits of the liquid phases are sometimes too conservative; *e.g.*, polypropylene glycols can be used up to at least 150° and not only up to 120° (p. 50). Some given values are contradictory to each other: *e.g.*, dimethyl sulfolane can be used at room temperature (p. 53) and not only up to 0° (p. 50).

When discussing the flame ionization detector (p. 74), it is incorrect that a H₂/N₂ mixture is used as carrier gas; the hydrogen is usually mixed only with the column effluent. Besides this one question, however, the author was very successful in the short compilation of the detectors, and particularly of the ionization detectors; this is the best part of the book. I just want to correct on p. 69 the reference to Fig. 4.1 which is a printing error: It should read, Fig. 4.2.

I could not understand why a T-shape construction is recommended for the sample splitter with the open tubular columns (p. 95 and Fig. 5.8): It has been proved in the literature and practice that this construction of the splitting device gives usually a non-linear splitting.

Finally, I would like to make two general remarks. The first refers to the symbols used in the book. It is very unfortunate that many of them not only differ from the recommended I.U.P.A.C. nomenclature, but some are even used in a quite different meaning. The most disturbing is the use of K as the capacity coefficient (ratio) while in most other publications, as well as in the I.U.P.A.C. nomenclature, K is reserved for the partition coefficient and k is used for the capacity ratio. At the same time, the book is using α as the partition coefficient (instead of K), although in other publications, α is usually the symbol for the relative retention. It would be most welcome if the different publications would agree on generally accepted symbols.

My second remark concerns the references. I understand and accept the intentions of the author not to give a detailed literature survey but only a few references after each chapter as selected literature (there are altogether only 43 references in the whole book). However, even so, some other publications should definitely be quoted: for example, those of Giddings in the theoretical part or the results of Bayer at the discussion of preparative columns. Besides this, it is interesting to note that although