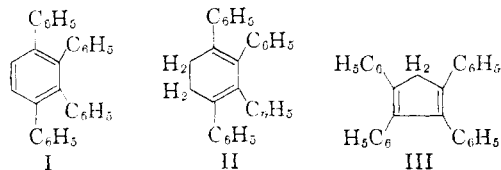


Found: C, 93.87; H, 6.07] is the product. This formulation is supported chemically by bromination of II with N-bromosuccinimide or bromine and dehydrobromination, and by dehydrogenation of II with palladium-charcoal, to 1,2,3,4-tetraphenylbenzene. The ultraviolet spectrum of II, having absorption maxima at 236 m μ , 268 m μ , and 326 m μ ($\epsilon = 15,600, 10,600$ and $12,100$) is also consistent with the cyclohexadiene structure. It is anticipated that the foregoing reaction will prove to be a general as well as a convenient method for preparing cyclohexadiene ring systems.



Mixed condensation between alkyl groups and acetylenes is shown, perhaps even more strikingly, by the reaction of trimethylchromium with toluene. In this example the organochromium(III) reagent contributes a methyl group, forming 1,2,3,4-tetraphenylcyclopentadiene, III, m.p. 176–178°,⁴ in addition to the normal product, hexaphenylbenzene. The oxidative potentialities of organochromium(III) are being examined in other areas of organic synthesis.

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RECEIVED OCTOBER 12, 1959

ACTIVE PHOSPHORUS

Sir:

An early attempt to produce phosphorus atoms by a condensed discharge proved unsuccessful.¹ In view of the work of Winkler and co-workers²

(1) N. M. Gopshtein and S. Z. Roginskii, *J. Phys. Chem. (USSR)*, **7**, 587 (1936).

(2) D. A. Armstrong and C. A. Winkler, *J. Phys. Chem.*, **60**, 1100 (1956).

on the reactivity of active nitrogen it was felt that a comparative study with phosphorus might prove informative.

An apparatus similar to that used for nitrogen² was tried for phosphorus. Argon, as a carrier gas, was passed through a bulb containing phosphorus at a temperature from 25 to 100°. The flow rate of argon was about 50 micromole/sec. at a pressure of approximately 1 mm. in the reaction bulb. When only argon was allowed to flow through the discharge, no decomposition of reactants was observed. Similarly no reaction was observed when P₄ was allowed to mix with reactant with no discharge. A definite reaction was obtained between phosphorus, swept through the discharge with argon, and ethylene, propylene, butene-1, propane, methane, ammonia, and hydrazine. Phosphine was the major gaseous constituent produced in all cases. The reactions also were observed when a microwave discharge was used to generate the "active phosphorus."

Gas chromatographic analysis on the products of methane-active phosphorus reactions indicated at least six volatile products. Phosphine, methylphosphine and ethane were obtained in very small quantities. The ethane was approximately 2% of the phosphine fraction. A considerable amount of solid was deposited in the reaction zone during an experiment. This, along with red phosphorus, appears to be the solid phosphorus hydrides.

It also was found that with a new discharge tube and a clean-walled apparatus, a liquid was produced in relatively large quantities. It was shown not to be P₂H₄ from physical properties and elementary reactions.

Further work along with the identification of the "liquid" product is now being pursued.

Grateful acknowledgment is made to the Research Corporation for supporting this work.

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RECEIVED OCTOBER 9, 1959

BOOK REVIEWS

Brandlehre und Chemischer Brandschutz. Eine Einführung in die Grundlagen. Zweite Ergänzte und Erweiterte Auflage. By LUDWIG SCHEICHL, Oberregierungsrat. Dr. Alfred Hüthig Verlag, Wilckenstrasse 3, Heidelberg, Germany. 1958. xiv + 424 pp. 16 × 23 cm. Price, DM 28.—.

This volume is not a scientific and technological treatment of ignition, combustion and explosions, as one would expect it to be if it were a technical book on "Brandlehre," but rather has as its main objective classifications and descriptions of materials in the technology of "Brandenschutz," i.e., of fire-proofing, fire-prevention, control and extinction. It would, therefore, have been more descriptive of the contributions of this book if the word "Brandlehre" had been omitted from the title. While technological aspects and classifications in "Chemischer Brandschutz" are well presented in the last two parts (Teil B and C) of the book, the first 207 pages, exclusive of 40 pages of classi-

fications and definitions pertaining to "Brandlehre" (pages 19–40 and 181–207) comprise good elementary, but unnecessary, reviews of well-known principles of physical chemistry. Pages 19 to 40 present interesting, but not actually fundamental, classifications of materials of combustion and definitions pertaining to ignition, flames, combustion and explosion. Pages 181 to 207 outline and classify burning processes and mechanisms, but the classifications are incomplete as are the descriptions of burning processes. It is true, however, that attention is called occasionally elsewhere in "Teil A" to applications of particular physico-chemical principles in combustion technology.

The actual fundamental principles comprising the science and technology of ignition, combustion and even fire-proofing and the control and extinguishing of combustion are merely mentioned briefly in spite of a great deal of the book being devoted to principles one might readily apply in this technology. It is interesting in view of the many

different topics of physical chemistry treated that several branches of physics and chemistry of special interest in combustion, explosion and detonation are not even mentioned, *e.g.*, propellant technology, the physics of rocketry, aerodynamics, hydrodynamics, and the physics and chemistry of ionized gases. The dearth in this volume of fundamental and detailed discussions and descriptions of ignition, flames, combustion, explosion and detonation is disappointing, especially in light of these extensive reviews, the title and subtitle, "Eine Einführung in die Grundlagen," the objectives stated in the preface of the book, and the tremendous general interest in these matters in our present era of propellants, rocketry, satellites, and space technology. It is, moreover, disappointing that practically none of the general principles discussed is applied specifically either to combustion technology or "Brandschutz." One may wonder, therefore, why so much of the book was devoted to these general subjects, *e.g.*, the electron structure of atoms, thermodynamics, reaction kinetics and the fundamentals of rate processes, the physics of radiation and absorption, Gibbs adsorption and certain aspects of atomic physics. All of these topics are, of course, available in excellent treatments elsewhere whether in elementary form for the beginner, more detailed and descriptive treatments for more advanced students, or comprehensive treatises for specialists. Certainly, one would not object to such introductory treatments, had they been applied directly to the particular technology treated in the volume.

The interests of this reviewer have not extended a great deal to considerations of ways and means of fireproofing substances, and controlling or extinguishing fires. The elementary considerations of the last two sections of this book treating "Brandschutz" were therefore of considerable interest to him and probably would be also to others whose main interests are combustion and explosives, and perhaps to scientists in general. In this light the parts of the book pertaining to this subject are valuable contributions to our scientific literature.

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M. A. COOK

Laboratory Distillation Practice. By E. A. COULSON, M.A., D.Sc. and E. F. G. HERINGTON, D.Sc., A.R.C.S. Interscience Publishers, Inc., 250 Fifth Avenue, New York 1, N. Y. 1958. x + 166 pp. 14 × 22 cm. Price, \$4.50.

This small book takes a useful practical non-theoretical approach toward its subject. Within this scope the treatment is quite complete, including columns and packings, associated equipment, simple theory, selection of operating conditions and practical considerations. The usual treatment on determination of the height of a theoretical plate is given, and a specific distillation unit is described in detail. Separate chapters deal with low temperature, low pressure, azeotropic distillation, measurement of vapor liquid equilibria, continuous laboratory distillation, extractive distillation, stripping, and fractionation of a reactive mixture. The discussion of azeotropes is more extensive than would be expected in a book of this kind. The British background of the authors is apparent in spots, such as the omission of any mention of widely used protruded packing. Some of the descriptions of specific pieces of equipment will be of limited utility because the devices mentioned are available only in the authors' country. Some packings of lesser importance and utility are included. The figures are excellent. The references are extremely limited in number and scope. The presentation is almost uniformly clear and suited particularly for new or occasional users of distillation techniques. The book is not intended for those already well versed in the subject. Items and subjects whose treatment is unusually good are manostats, thermocouple junction welding and the explanation and nomograph of the Fenske equation. The discussion on boilers and boilup rate measurement is weak. There might have been more emphasis on the increase in HETP with diameter in a packed column, a mention of the desirability of a holding device at the top of a packed column, a mention of electric heating mantles, and more about head temperature measurement and test mixtures. Nevertheless every laboratory that has occasion to do any distillation whatever should have this book at

hand, and time spent in reviewing its clear discussions will be much more than saved in improved apparatus and operation.

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The Chemistry of Natural Products. Volume II. Mono- and Sesqui-terpenoids. By P. DE MAYO, Imperial College of Science and Technology, London, England. K. W. Bentley, Editor. Interscience Publishers, Inc., 250 Fifth Avenue, New York 1, N. Y. 1959. vii + 320 pp. 16 × 23.5 cm. Price, \$7.50.

The Chemistry of Natural Products. Volume III. The Higher Terpenoids. By P. DE MAYO, Imperial College of Science and Technology, London, England. K. W. Bentley, Editor. Interscience Publishers, Inc., 250 Fifth Avenue, New York 1, N. Y. 1959. vii + 239 pp. 16 × 23.5 cm. Price, \$6.00.

The original purpose of this series of books under the general editorship of K. W. Bentley is to provide reasonably priced and reasonably sized introductory monographs to various areas of natural products chemistry. The two volumes under review are intended to cover the terpene field. This should be particularly welcome among American academic circles, where this subject is greatly neglected. Indeed, de Mayo's preface to these two volumes should be compulsory reading, because it states succinctly and convincingly the case for the enormous pedagogic and scientific value of terpenoids. The general approach is indicated in the preface by an apt quotation from Milne's *Winnie-The-Pooh* and the fact that the author has not succeeded completely is due largely to his publisher and to a certain extent also to unfortunate timing. If these books had been written toward the latter part of 1958 rather than in early 1957, they could have covered a very considerable number of recent advances, but more about this later.

To quote again A. A. Milne, "nobody—nobody could call me a fussy man BUT" this reviewer considers it unforgivable that two short books of this type took the publisher two years to produce. This is particularly preposterous in this instance where all structural formulas have been drawn by hand (at times a rather shaky one) and reproduced directly. Such books could be issued in less than six months or else additions should be made in proof to bring them up to date. In the final analysis, it is up to authors to bring pressure upon publishers to reduce the time interval to a reasonable limit, and this certainly was not done here. As a result, several recent advances are not included and this is precisely the material which would make the uninitiated student and his professor look up and pay notice. All of the classical studies can be found in Simonsen's "The Terpenes" and other compendia; the purpose of a short introductory book should, therefore, not be a condensation of the historical literature but rather to emphasize the most recent advances, using modern approaches, and to place the earlier literature (without disrespect, indeed often with admiration) into its proper perspective. With one unfortunate exception, de Mayo attempted and largely succeeded in accomplishing this (especially in Vol. II) and it is a pity that circumstances prevented him from carrying the literature coverage to late 1958 as could so easily have been done.

The introductory chapter aptly contains reference to various physical methods which have been used so extensively in recent times in structure studies of terpenoids. The coverage of ultraviolet and infrared methods is quite adequate, but immediately thereafter, the long publication time interval or the unfortunate timing becomes apparent. There is very brief mention of nuclear magnetic resonance, even less of rotatory dispersion and as far as the reviewer could determine, none of mass spectrometry. The last omission may be due to modesty since one of the important papers has been published by de Mayo and Reed.

The discussion of monoterpenoids is on the whole very satisfactory, especially chapter 4 dealing with the intricate rearrangements of bicyclic monoterpenes through non-classical carbonium ions. The least satisfactory portion is the one dealing with absolute configurations, the two pages starting on p. 174 representing really only a reprint of Bireli's list (ref. 40). This will not teach a student how these stereochemical conclusions were really arrived at