

into water (200 ml) with stirring. The precipitate (dry weight, 436 mg) was collected by filtration and thoroughly washed with water. The amount of thymidine in this product was determined by treatment with trifluoroacetic acid (TFA) in chloroform (1:99, v/v) at 0°, under which conditions thymidine release was complete in under 5 min. The amount of thymidine in IV was thus determined to be 6 μ moles/100 mg of IV. By a similar procedure polymer-supported 5'-O-monomethoxytrityl-N-benzoyldeoxyadenosine was prepared from III, the adenine content being 2.9 μ moles/100 mg of the product.

For internucleotide bond synthesis, IV (190 mg) was allowed to react with pyridinium 3'-O-acetylthymidine 5'-phosphate (pT-OAc, 80 mg) and mesitylene sulfonyl chloride (77 mg) in dry pyridine (2.5 ml) at room temperature for 3.5 hr. (More pyridine (2 ml) was added after 1.5 hr.) After treatment with water (0.4 ml) and pyridine (2.5 ml) at room temperature for 15 hr, the clear reaction solution was poured into 500 ml of 2% aqueous sodium chloride solution. The precipitate was collected by filtration, washed with water, and dried over phosphorus pentoxide. The 3'-O-acetyl group was removed with 1 M sodium methoxide in methanol-dimethyl sulfoxide-pyridine (1:5:4, v/v, 20 ml; 15 min at room temperature). The alkaline solution was poured into 500 ml of 2% aqueous sodium chloride and the resulting precipitate (178 mg) of V was collected by centrifugation followed by filtration and washing with water. Subsequent treatment with TFA-chloroform mixture followed by paper chromatographic analysis⁸ showed thymidylyl-(3'→5')-thymidine (TpT) and thymidine as the only products, the yield of TpT being 96%. In a similar manner, \oplus -d-TpC^{An}-OAc⁹ and

\oplus -d-TpC^{An}-OAc⁹ were synthesized in yields of 92 and 91%, respectively.

Repetition of the condensation of V (125 mg) with pT-OAc (200 mg) and mesitylenesulfonyl chloride (200 mg) in pyridine (2.5 ml) was performed at room temperature for 2.75 hr. Aqueous pyridine treatment at room temperature for 17 hr, precipitation in 2% aqueous sodium chloride, sodium methoxide treatment at room temperature for 12 min, and final precipitation in 2% aqueous sodium chloride yielded VI (105 mg). TFA-chloroform liberated mostly TpTpT (VII), some TpT, and a little T. TpTpT contained two trace contaminants (pT, 1.7%, and another nucleotide, 1.9%) as shown by subsequent electrophoresis. The yield of pure TpTpT from TpT was 87%.

All of the products, TpT, d-TpG, d-TpC, and TpTpT, were checked for their purity in paper chromatography (two solvents) and paper electrophoresis and were completely susceptible to the action of spleen phosphodiesterase, thus showing the exclusive presence of C₃-C_{5'} internucleotidic linkages in them.

The approach described incorporates all of the principles of the methods previously developed for construction of deoxyribopolynucleotide chains containing predetermined sequences. For example, the 5'-hydroxyl group of the terminal nucleoside is protected by a substituted trityl group and the chain elongation occurs by successive condensations with the terminal 3'-hydroxyl group. An important requirement is that yield at every repetition of internucleotide bond synthesis be as nearly quantitative as possible, and this appears to be the case in the several examples studied so far. Experiments on the use of this approach in the synthesis of longer oligonucleotide chains are in progress.

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(8) Using the solvent: 2-propanol-concentrated ammonia-water (7:1:2, v/v).

(9) Abbreviations for the protected deoxyribooligonucleotides are as defined previously: H. Shaller and H. G. Khorana, *J. Am. Chem. Soc.*, **85**, 3841 (1963).

Book Reviews

An Introduction to Mathematical Crystallography. By M. A. JASWON, M. A., Ph.D., Reader in Mathematics, Imperial College, London. American Elsevier Publishing Co., Inc., 52 Vanderbilt Ave., New York, N. Y. 1965. xi + 125 pp. 14.5 × 22 cm. \$6.00.

The term "Mathematical Crystallography" in the title of this book is taken by the author to refer to the analysis of the symmetry properties of periodic structures in three dimensions, *i.e.*, the 32 point groups and the 230 space groups. In the preface the author states, "Although the theory was completed by 1890, and no new results can be expected, considerable scope still remains for fresh presentations and interpretations." He also states that, "The approach... steers a middle course between excessive formalism and excessive perspective geometry...".

In the course of 103 pages plus 21 pages of appendices, the author develops the point groups and space groups in a more or less systematic manner. After a fairly normal introduction of the point groups he develops the space groups from the "motif" point of view of Bravais. This took courage on the part of the author since it was perhaps this point of view which held back the development

of the complete theory of space groups for about 40 years. It does give one a mass of material on the arrangement of point group replicas in space. From this material he develops the ideas of screw axes and glide planes, using an interesting notation for the treatment of translation operators. His discussion of the "diamond glide" space groups is very brief and probably needs a good deal of expansion to make it understandable to a student.

The chief difficulty the reviewer has had in understanding this book lies in the way that the author has chosen the "middle course" referred to above. He has given a development of the point groups "based upon intuitive geometrical considerations." This, one has to do, but to omit the basic postulates and theorems of group theory from this book on the grounds that they "may be found in numerous texts" is inexcusable.

One is grateful that the author has used the Hermann-Mauguin notation for the point and space groups which has been adopted internationally for 30 years. It is however somewhat unfortunate that the operation $\bar{3}$ is first presented in this book as a sixfold rotatory reflection and not as a threefold rotatory inversion as was intended by those who developed the notation.

The most serious pedagogical mishap seems to the reviewer to occur in Chapter 8, Table 4, which is headed "Enumeration of the sixty-six Bravais space groups." In this table the author indicates that each of the 32 point groups can be laid down on the 14 Bravais lattices giving the count of 66 "Bravais" space groups. It seems very wrong to give prominence to this number 66 and then to devote the following pages to demonstrate that seven of the point groups can be laid down in two different ways on the corresponding lattices to bring the total to 73.

There is much of value in this book, and it should be read by anyone who is planning a serious course of space-group theory, and it could perhaps be used as a textbook to accompany the lectures of such a course. But it cannot be recommended for use by a beginner working alone.

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Physical Methods in Organic Chemistry. Edited by J. C. P. SCHWARZ, M.A., B.Sc., Ph.D., Lecturer in Chemistry, University of Edinburgh, Edinburgh, Scotland. Holden-Day, Inc., 728 Montgomery St., San Francisco, Calif. 1965. xi + 350 pp. 15 × 23 cm. \$9.75.

The use of physical methods by the organic chemist for structure determinations is becoming increasingly widespread. As a result, there is a need for a single-volume book that provides a critical and instructive introduction to the methods most commonly employed. This edited volume attempts to provide a readable introduction to such physical methods. This it does. The contributed chapters provide the reader with the scope of the physical method under discussion together with ample references to key papers, review articles, and monographs. With several exceptions, the contents of the chapters go little beyond the elementary level, and, despite a fair number of well-selected examples and tabulated empirical correlations, they will leave the serious reader distinctly dissatisfied. No wonder, for a total of eight physical methods are presented in only 300 pages—about 100 of which are devoted to infrared spectroscopy, leaving the remainder for coverage of seven other topics. Clearly, the book is too short for such a broad coverage. Consequently, it will be of limited service, except as a guide to the literature, to the practicing organic chemist.

In Chapter 1, the editor provides the reader with a useful survey of the methods to be discussed in the remaining chapters. Chapter 2 (by Bladon and Eglinton) is a short, but useful, outline of the common features of ultraviolet, visible, and infrared spectroscopy. Chapter 3 (by Eglinton) which comprises over one-fourth of the book, discusses infrared, and, to the extent of several pages, Raman spectroscopy. This is the best elementary introduction to infrared spectroscopy that this reviewer has seen. It is packed with useful information and examples of applications. Chapters 4 (by Bladon), 5 (by Jackman), and 6 (by Schwarz) by comparison are rather weak introductions to ultraviolet and visible spectroscopy, magnetic resonance spectroscopy (primarily nmr with a few pages devoted to esr), and optical rotation (ORD, CD, and single-wavelength rotations), respectively. Better introductions to these topics can be found elsewhere. Chapter 7 (by Greenwood) is an excellent critical discussion of methods for determining molecular weights ranging from low molecular weight monomers to high molecular weight polymers. Chapters 8 (by Sim), 9 (by Reed), and 10 (by Shaw) too briefly cover the topics of diffraction methods (exclusively X-ray), mass spectrometry, and dipole moments, respectively.

This book is not sufficiently self-contained to be of value as a text for an introductory course in physical methods. It may be useful to the organic chemist as a cursory introduction to the physical methods described and as a guide to more comprehensive works. All organic chemists would profit by reading Eglinton's chapter on infrared and Raman spectroscopy.

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