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### PHYSICAL METHODS of Organic Chemistry

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TECHNIQUE OF ORGANIC CHEMISTRY ARNOLD WEISSBERGER, Editor

Volume 1 PHYSICAL METHODS OF ORGANIC CHEMISTRY Part Three Second Completely Revised and Augmented Edition

### TECHNIQUE OF ORGANIC CHEMISTRY ARNOLD WEISSBERGER, Editor

Physical Methods of Organic Chemistry  $Volume\ I$ :

Second Edition

Parts I to III

Catalytic, Photochemical, and Electrolytic Volume II:

Reactions

 $Volume\ III:$ 

Heating and Cooling; Mixing; Centrifuging; Extraction and Distribution; Dialysis and Electrodialysis; Crystallization and Recrystallization; Filtration; Solvent Re-

moval, Evaporation, and Drying

Volume IV: Distillation

Volume V: Adsorption and Chromatography

Volume VI: Micro and Semimicro Methods

Volume VII: Organic Solvents

Second Edition

Volume VIII: Investigation of Rates and Mechanisms of

Reactions

Volume IX: Chemical Applications of Spectroscopy



# PHYSICAL METHODS of Organic Chemistry

Second Completely Revised and Augmented Edition

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PART THREE

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INTERSCIENCE PUBLISHERS, INC., NEW YORK INTERSCIENCE PUBLISHERS LTD., LONDON



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INTERSCIENCE PUBLISHERS, INC. 250 Fifth Avenue New York 1, N. Y.

For Great Britain and Northern Ireland: INTERSCIENCE PUBLISHERS LTD. 88/90 Chancery Lane London W. C. 2

PRINTED IN THE UNITED STATES OF AMERICA BY MACK PRINTING CO., EASTON, PA.

### TECHNIQUE OF ORGANIC CHEMISTRY

#### INTRODUCTION

Organic chemistry, from its very beginning, has used specific tools and techniques for the synthesis, isolation, and purification of compounds, and physical methods for the determination of their properties. Much of the success of the organic chemist depends upon a wise selection and a skillful application of these methods, tools, and techniques, which, with the progress of the science, have become numerous and often intricate.

The present series is devoted to a comprehensive presentation of the techniques which are used in the organic laboratory and which are available for the investigation of organic compounds. The authors give the theoretical background for an understanding of the various methods and operations and describe the techniques and tools, their modifications, their merits and limitations, and their handling. It is hoped that the series will contribute to a better understanding and a more rational and effective application of the respective techniques. Reference is made to some investigations in the field of chemical engineering, so that the results may be of assistance in the laboratory and help the laboratory chemist to understand the problems which arise when his work is stepped up to a larger scale.

The field is broad and some of it is difficult to survey. Authors and editor hope that the volumes will be found useful and that many of the readers will let them have the benefit of their criticism and of suggestions for improvements.

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### TECHNIQUE OF ORGANIC CHEMISTRY

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- Volume II. Catalytic Reactions, V. I. Komarewsky and C. H. Riesz; Photochemical Reactions, W. A. Noyes, Jr., and V. Boekelheide; Electrolytic Reactions, S. Swann, Jr.
- Volume III. Heating and Cooling, R. S. Egly; Mixing, J. H. Rushton and M. P. Hofmann; Centrifuging, H. B. Golding; Extraction and Distribution, L. C. Craig and D. Craig; Dialysis and Electrodialysis, R. E. Stauffer; Crystallization and Recrystallization, R. S. Tipson; Filtration, A. B. Cummins; Solvent Removal, Evaporation, and Drying, G. Broughton
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- Volume VI. Micro and Semimicro Methods. N. D. Cheronis. With contributions by A. R. Ronzio and T. S. Ma
- Volume VII. Organic Solvents. A. Weissberger and E. S. Proskauer. Second Edition by J. A. Riddick and E. E. Toops, Jr.
- Volume VIII. Investigation of Rates and Mechanisms of Reactions. Editors: S. L. Friess and A. Weissberger. Contributors: G. M. Burnett, B. Chance, E. Grunwald, S. L. Friess, F. M. Huennekens, T. H. James, T. S. Lee, J. E. Leffler, R. Livingston, H. W. Melville, B. K. Morse, P. R. O'Connor, W. J. Priest, F. J. W. Roughton, and W. D. Walters
- Volume IX. Chemical Applications of Spectroscopy. Editor: W. West. Contributors: A. B. F. Duncan, W. Gordy, R. Norman Jones, F. A. Matsen, C. Sandorfy, and W. West

### TECHNIQUE OF ORGANIC CHEMISTRY Volume I

### PHYSICAL METHODS OF ORGANIC CHEMISTRY

From the Prefaces to the First and Second Editions of Parts I and II

In recent years, the science of physics has become increasingly important to the organic chemist. Physics has given much greater precision to the concept of atoms, bonds, and structural formulas, and it has made possible the development of new, and the improvement of older, methods for the examination of chemical systems. With the increasing number and complexity of physical methods for the treatment of organic chemical problems there has resulted a specialization of research workers in the methods which they employ, and the selection of a research problem is frequently governed more by the physical method to be used than by the chemical nature of the problem. Some workers have made themselves familiar with several methods in order to deal with their individual problems. In other cases, however, physical methods have been used without adequate preparation.

The chemist, in order to acquaint himself with a certain physical method, has in the past been compelled to search through periodicals and specialized books. The present work has been compiled with the hope of relieving him of much of this burden. It has been the object of the authors to provide a description of tested methods, the theoretical background for understanding and handling them, and the information necessary for a critical evaluation of the experimental results.

Because of the diversity of the methods discussed, no attempt has been made to secure a uniformity of presentation which might have been desirable for formal reasons. In some chapters a discussion of theory was unnecessary, in some a relatively brief theoretical treatment sufficed, and in other chapters a rather complete exposition of the theory appeared necessary. Some methods have been treated in monographs, while for others no comprehensive modern presentation is available. Therefore, a rather severe selection and delimitation of material was exercised in some chapters and a more complete treatment given in others.

The book is also calculated to appeal to the student who seeks to increase his understanding of the methods described, although he may not practice them himself. For him, chapters like those on x-ray and electron diffrac-

tion should be adequate, but the practical application of these techniques will require the use of the supplemental literature to which reference is made.

The title, Physical Methods of Organic Chemistry, has been called too narrow. "Organic Chemistry" distinguishes the methods described from those physicochemical methods which, though essential in other fields, are less important for, or not applicable to, organic chemistry—for example, methods employing very high temperatures. We do not, of course, suggest that the methods described are applicable to organic problems only. Though it is gratifying that workers in other fields have found the book useful, it is our chief object to provide information on the physical methods used by chemists, physicochemists, physicists, biologists, and other research workers in dealing with organic chemical problems.

A. W.

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### TECHNIQUE OF ORGANIC CHEMISTRY Volume I

### Preface to Part III

The first edition of this treatise appeared in 1945 (Part I) and 1946 (Part II). It was followed by a second edition in 1949 (Parts I and II); in this, new chapters were added and others were rewritten, substantially revised, or enlarged.

As can be expected in a rapidly expanding field, it has again become desirable to present newly developed methods, and a new edition was considered. Closer analysis, however, showed that large sections of the second edition of *Physical Methods of Organic Chemistry* do not require revision, and it did not seem right to link the new with the unchanged so that owners of the second edition would have to duplicate material on their shelves in order to gain access to the new and the revised chapters. The second edition is, therefore, expanded and rejuvenated by the present Part III rather than made obsolete by a new edition.

Physical Methods of Organic Chemistry, Part III, contains the following new chapters in fields not treated or only touched upon in the first two parts: Electron Microscopy, by F. A. Hamm; Microspectroscopy, by E. R. Blout; Determination of Streaming Birefringence, by R. Signer; Measurement of Dielectric Constant and Loss, by J. G. Powles and C. P. Smyth; Radio-frequency Spectroscopy, by B. P. Dailey; and Neutron Diffraction, by J. M. Hastings and L. Corliss. Additions to chapters contained in Part II have been supplied by T. E. McGoury and H. Mark, Viscometry of Dilute Polymer Solutions; D. Harker, Determination of Crystal Structure of Organic Compounds by X-Ray Diffraction; L. O. Brockway, Electron Diffraction by the Sector-Microphotometer Method; J. F. Bonner, Determination of Radioactivity by Scintillation Counting; and a new presentation of Determination of Magnetic Susceptibility has been contributed by P. W. Selwood.

A. W.

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