Volume I—Part I

PHYSICAL METHODS of Organic Chemistry

Third Completely Revised and Augmented Edition

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ARNOLD WEISSBERGER

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PART ONE



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Reactions

Second Edition

Volume III: Second Edition

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Volume VI: Micro and Semimicro Methods

Volume VII: Organic Solvents

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Volume VIII: Investigation of Rates and Mechanisms of

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Volume IX: Chemical Applications of Spectroscopy

Volume X: Fundamentals of Chromatography



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.

A. W.

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Volume I

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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 diffraction 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

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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.

Preface to the Third Edition

The Second Edition of this treatise was published in 1949 and brought up to date by Part III in 1954. Since then the continued high demand for Parts I and II has indicated the need for either a reprinting or a new edition, and a survey has shown that only a new edition would cope with the changes in, and the additions to, the rapidly developing field.

In the Third Edition of *Physical Methods* new topics have been added, as chapters on "Automatic Control" by J. M. Sturtevant, "Automatic Recording" by D. R. Simonsen, "Weighing" by A. Corwin, "Determination of Particle Size and Weight" by G. Beyer, "X-Ray Microscopy" by W. Nixon, "Determination of the Kerr Effect" by R. J. W. and C. G. Le Fevre, "Determination of the Faraday Effect" by C. E. Waring and R. Custer, "Nuclear Magnetic Resonance" by H. S. Gutowsky, "Electron Spin Resonance" by G. K. Fraenkel, "Determination of Transference Numbers" by M. Spiro, and "Coulometric and Related Methods" by L. Meites.

Automatic control and automatic recording are applicable to many of the methods described in the treatise, and the systematic discussions of Chapters I and II obviate repetitious references. Weighing, likewise, is of general importance. Particle size is influential on, and determined by means of, measurements with so many different methods that a unifying survey appeared desirable. Other new chapters take care of omissions in the earlier editions and of recent additions to the arsenal of methods which are applicable to organic compounds.

The two chapters on "Spectroscopy and Spectrophotometry" and on "Colorimetry, Photometric Analysis, Fluorimetry, and Turbidimetry" by W. West have been divided into five chapters: "Visible and Ultraviolet Spectroscopy and Spectrophotometry" by W. West, "Infrared Spectrophotometry" by D. Anderson, W. West, and N. B. Woodall, "Colorimetry and Photometric Analysis" by W. West, "Determination of Fluorescence and Phosphorescence" by G. Oster and N. Wotherspoon, and "Determination of Light Scattering" by G. Oster. The importance of "Optical

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Rotatory Dispersion" is reflected in a chapter by W. Klyne and A. C. Parker which supplements the chapter on "Polarimetry."

We deeply regret the death of Dr. W. D. Harkins, Dr. L. Michaelis, and Dr. M. A. Peacock. Their topics have been taken over by Dr. A. E. Alexander, "Determination of Properties of Insoluble Monolayers at Mobile Interfaces" and "Determination of Surface and Interfacial Tension," Dr. J. D. H. Donnay and Dr. Gabrielle Donnay, "Determination of Crystal Morphology," Dr. P. W. Selwood, "Determination of Magnetic Susceptibility," and Dr. C. Tanford and Dr. S. Wawzonek, "Potentiometry." Where substantial parts of the original chapters were retained, credit is given in the titles to the former authors.

Dr. S. Z. Lewin, Dr. J. Arthur, Dr. W. J. Mader, Dr. J. F. Swindells, Dr. R. Ullman, Dr. L. D. Moore, Dr. R. Pontius, Dr. G. Donnay, Dr. W. N. Lipscomb, Dr. D. D. Fitts, Dr. H. C. Scheraga, Dr. B. M. Tolbert, and Dr. W. Siri have joined or replaced authors of the earlier editions in the writing of the chapters, "Determination of Density" by N. Bauer and S. Z. Lewin, "Determination of Melting and Freezing Temperatures" by E. Skau and J. Arthur, "Determination of Solubility" by W. J. Mader, R. D. Vold, and M. J. Vold, "Determination of Viscosity" by J. F. Swindells, R. Ullman, and H. Mark, "Determination of Osmotic Pressure" by R. H. Wagner and L. D. Moore, "Determination of Diffusivity" by A. L. Geddes and R. Pontius, "Refractometry" by N. Bauer, S. Z. Lewin, and K. Fajans, "Crystallochemical Analysis" by J. D. H. Donnay and G. Donnay, "X-Ray Diffraction" by W. N. Lipscomb, "Polarimetry" by W. Heller and D. D. Fitts, "Determination of Streaming Birefringence" by H. S. Scheraga and R. Signer, and "Determination of Radioactivity" by B. M. Tolbert and W. Siri.

Some physical methods have been omitted from this volume because they are discussed elsewhere in the Series. Certain techniques useful in preparative work are treated in Volume II, Catalytic, Photochemical, and Electrolytic Reactions, and in Part I of Volume III, Separation and Purification. Additional information on vapor pressure and volatility may be found in Volume IV, Distillation. Special techniques for work on a micro and semimicro scale are presented in Volume VI. Adsorption and Chromatography are treated in Volumes V and X, respectively.

The application of certain of the methods discussed in the present treatise to the study of rates and mechanisms of reactions is elaborated in Volume VIII. Chemical Applications of Spectroscopy are the topic of Volume IX, but applications of the non-spectroscopic physical methods are discussed in the present volume in order to explain the use and the scope of the respective techniques.

In sub-dividing the treatise into Parts I to IV we have tried not to separate

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closely related topics. Individual indices summarize the contents of Parts I, II, and III, and an index of the whole treatise is contained in Part IV.

I am grateful to many colleagues for help in the editorial work; to Dr. E. K. Blout, Dr. H. S. A. Gilmour, Dr. T. Gompf, Dr. R. L. Griffith, Dr. F. Hamm, Dr. G. Happ, Dr. A. Herz, Dr. W. J. Knox, Jr., Dr. W. Moffitt, Dr. O. H. Muller, Mr. D. Pearlman, Dr. R. Pontius, Dr. B. W. Rossitter, Dr. W. R. Ruby, Dr. H. Seeman, Dr. D. R. Simonsen, Dr. W. R. Vaughan, and to my wife, Dr. Louise H. Weissberger. Last, but not least, I want to express my gratitude to the publishers and their staff, not only for their expert handling of technical matters, but for their willingness to undertake a new edition instead of a less onerous, more remunerative, but for the user less satisfactory, reprinting of the second edition.

A. W.

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