



TECHNIQUE OF ORGANIC CHEMISTRY
ARNOLD WEISSBERGER, *Editor*

Volume III

Second Completely Revised and Augmented Edition

PART I. SEPARATION AND PURIFICATION

TECHNIQUE OF ORGANIC CHEMISTRY
ARNOLD WEISSBERGER, *Editor*

- Volume I.* Physical Methods of Organic Chemistry
Second Edition
Parts I to III
- Volume II.* Catalytic, Photochemical, and Electrolytic Reactions
Second Edition
- Volume III.* *Second Edition*
Part I Separation and Purification
Diffusion Methods Laboratory Extraction and Countercurrent Distribution
Crystallization and Recrystallization
Centrifuging Filtration Solvent Removal, Evaporation, and Drying
Part II Laboratory Engineering Selection of Materials for the Construction of Equipment Heating and Cooling Grinding, Screening, and Classifying Mixing Operations with Gases
- 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



PART I
SEPARATION AND
PURIFICATION

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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.

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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TECHNIQUE OF ORGANIC CHEMISTRY

Editor ARNOLD WEISSBERGER

GENERAL PLAN

Volume I (Second Edition—in three parts). Physical Methods of Organic Chemistry. J R Anderson, E D Bailey, W F Bale, N Bauer, E R Blout, J F Bonner, Jr, L O Brockway, L Colless, B P Dailey, J D H Donnay, K Fajans, I Fankuchen, A L Geddes, F A Hamm, D Harker, W D Harkins, J M Hastings, W Heller, E E Jelley, T E McGoury, H Mark, L Michaelis, D H Moore, O H Muller, J B Nichols, M A Peacock, J G Powles, P W Selwood, T Shedlovsky, R Signer, E L Skau, C P Smyth, D W Stewart, J M Sturtevant, W Swietoslowski, G W Thomson, M J Vold, R D Vold, R H Wagner, H Wakeham, and W West

Volume II (Second Edition). Catalytic Reactions, V I Komarewsky, C H Riesz, and F L Moritz, **Photochemical Reactions,** C R Masson, V Boekelheide and W. A. Noyes, Jr, **Electrolytic Reactions,** S Swann Jr

Volume III (Second Edition).

Part I. Separation and Purification: Diffusion Methods, A Letcher Jones, K Kammermeyer, R E Stauffer, and E MacWilliam, **Laboratory Extraction and Countercurrent Distribution,** L C Craig, D Craig, and E G Scheibel, **Crystallization and Recrystallization,** R S Tipson, **Centrifuging,** C M Ambler and F W Keith Jr, **Filtration,** A B Cummins and F B Hutto, Jr, **Solvent Removal, Evaporation, and Drying,** G Broughton

Part II. Laboratory Engineering: Selection of Materials for the Construction of Equipment, R F Eisenberg and R R Kraybill **Heating and Cooling,** R S Egly, **Grinding, Screening, and Classifying,** J W Axelson and W C Streib, **Mixing,** J H Rushton and M P Hofmann, **Operations with Gases,** G H Miller

Volume IV. Distillation. J R Bowman, C S Carlson, A L Glasebrook, J C Hecker, E S Perry, Arthur Rose, E Rose, R S Tipson, and F E Williams

Volume V. Adsorption and Chromatography. H G Cassidy

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 *Authors* 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 Moies, P R O'Connor, W J Priest, F J W Roughton, and W D Walters

Volume IX. Chemical Applications of Spectroscopy. *Editor* W West *Authors* A B F Duncan, W Gordy, R Norman Jones, F A Matsen, C Sandorfy, and W West

PREFACE TO THE SECOND EDITION

The first edition of this volume contained, in a rather loose arrangement, chapters dealing with general methods and operations used in preparative organic chemistry. Distillation, Adsorption and Chromatography were omitted because Volumes IV and V, respectively, are devoted to these techniques, and these methods are for the same reasons not included in the new edition of Volume III.

In this edition, methods of *Separation and Purification* used in preparative organic chemistry have been collected in a special *Part I*. The older chapters were revised or rewritten, and new sections were added to deal with Thermal Diffusion, Barrier Separation, Zone Electrophoresis, Liquid-Liquid Extraction for Increased Quantities, Inclusion Complexes, and other topics not treated in the first edition.

The chapters on Heating and Cooling and on Mixing, in the first edition, dealt with techniques long recognized in their importance for pilot plant operation and large-scale production but often handled haphazardly in the laboratory. These presentations have been found useful in the design and the operation of laboratory equipment, particularly on a larger scale. Both chapters were brought up to date for the new edition and the chapter on Heating and Cooling was in part rewritten and expanded, emphasizing practical applications to supplement the chapters by J. M. Sturtevant in Volume I of this series. The success of these two chapters has encouraged the addition of new chapters of a similar nature dealing with Choice of Materials for Equipment, with Diminution and Classifying, and with Operations with Gases, and to collect these chapters in a special *Part II on Laboratory Engineering*.

We deeply regret the death of two of the authors of the first edition, Mr. H. Golding and Dr. G. Broughton. Dr. C. M. Ambler has taken over the authorship of the chapter on Centrifuging which was written for the first edition by the late H. Golding. I am grateful to Dr. S. Miller, successor to the late Dr. G. Broughton, and to Miss S. Stamin for reading the proof of the chapter on Solvent Removal, Evaporation, and Drying which Dr. Broughton completed in manuscript form. To

Mr J W Axelson and Drs G. Beyer, C. Duboc, F. Kottler, E A MacWilliam, E Perry, R E Stauffer, and F. Urbach, I owe thanks for advice and assistance in various phases of the editorial work

A W.

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