VOGEL'S TEXTBOOK OF PRACTICAL ORGANIC CHEMISTRY

including

QUALITATIVE ORGANIC ANALYSIS

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QUALITATIVE ORGANIC ANALYSIS

Fourth edition
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Thames (formerly Woolwich) Polytechnic

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including Qualitative Organic Analysis

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PREFACE TO FOURTH EDITION

It is now twenty-eight years since the first appearance of Vogel's *Textbook of Organic Chemistry* and twenty years since the publication of the previous edition, and it is a tribute to the thoroughness and farsightedness of the late Dr A. I. Vogel that the book continues to be used as a standard text in the organic chemistry laboratory.

The period since the appearance of the third edition has seen considerable changes in the practice and theory of organic chemistry. Among these may be included the ready availability of a much wider range of substrates and reagents; the development of a whole host of new synthetic methods; a greater awareness of the hazards associated with handling of organic chemicals; the now routine use of chromatographic and spectroscopic methods; and the use of mechanistic concepts to rationalise and predict the outcome of organic reactions. In preparing this new edition it has been our intention to reflect these changes in the subject whilst at the same time maintaining the essential character of the work, which has become an invaluable one-volume reference text both for undergraduates and postgraduates, and for the practising organic chemist.

The text has been completely revised in the light of current theory and practice. Major sections have been completely rewritten or reorganised and a substantial amount of new material has been added. In order to keep the size of the volume to realistic proportions it has been necessary to make room for much of the new material by discarding some of the previous contents which have either been superseded, or are not now directly relevant to the practising chemist. The major casualty in this respect was the chapter dealing with the Theory of General Technique, but this is now available to the reader in the second edition of Vogel's *Elementary Practical Organic Chemistry*.

The book now commences with the chapter on Experimental Techniques which has been completely revised and is arranged under the following subheadings: Apparatus and Reaction Procedures (incorporating most of the special techniques previously in the chapter entitled Miscellaneous Reactions); Isolation and Purification Procedures; and the Determination of Physical Constants. Major introductions in this chapter are the Sections on Safe Working in Organic Chemical Laboratories (**I,3**), Chromatography (**I,31**) and Spectroscopic Methods (**I,39**). We have included examples where appropriate of the use of chromatographic techniques and of spectroscopic methods in some of the newly incorporated experimental sections. Aspects of the interpre-

tation of infrared, ultraviolet–visible, nuclear magnetic resonance and mass spectra are developed in Chapter VII, Qualitative Organic Analysis, so that the reader can gain experience in the use and appreciate the value of these techniques. Chapter I also includes an extended bibliography to allow the interested reader to obtain further information if required. Substantially expanded and up-dated Sections on the Purification of Common Organic Solvents, and the Preparation and Purification of Reagents now constitutes Chapter II.

The preparative sections are now organised into four chapters: III, Aliphatic Compounds; IV, Aromatic Compounds; V, Some Alicyclic Compounds; and VI, Some Heterocyclic Compounds. We have retained a substantial proportion of the reactions from the previous edition which continue to be of value either as examples for student exercises, or as standard preparative procedures. The examples included in the Alicyclic and Heterocyclic chapters are mainly restricted to methods for the formation of the cyclic system; any functionally group modifications in these systems are included in the appropriate sections of the other chapters. The preparations previously included in the chapters entitled Miscellaneous Reactions; Organic Reagents in Inorganic and Organic Chemistry; Dyestuffs, Indicators and Related Compounds; Some Physiologically Active Compounds, have now been incorporated as appropriate into the four new Chapters.

The past two decades have seen the publication in the literature of a vast number of new synthetic methods. We have sought in our selection of new preparative material to exemplify some of these new techniques and methods of synthesis where the value and generality has been well proven. It is not possible here to give a comprehensive list of all the additions; the following are some of the important reactions or reagents, examples of the use of which have been introduced into this edition: lithium dialkyl cuprates (Section **III,5**); phosphorus ylides (Section **III,14**); sulphur ylides (Section **VI,35**); formation of allenes (Sections **III,17** and **III,18**); selective oxidation (Sections **III,87** and **IV,25**); hydroboronation followed by oxidation (Sections **III,43** and **III,89**); selective reductions (Sections **IV,57**, **IV,65** and **VI,1**); acetylenic coupling reactions (Sections **III,22** and **III,23**); hydration of alkynes (Section **III,91**); photochemical reactions (Sections **III,30**, **IV,17**, **V,16** and **V,17**); oxymercuration-demercuration (Section **III,44**); use of aprotic solvents (Section **III,161**); enzymic resolution (Section **III,198**); benzyne intermediates (Section **IV,9**); use of triphenylphosphine dibromide (Section **IV,34**); formylation reactions (Section **IV,129**); esterification procedures (Sections **III,148** and **IV,179**); carbene and phase transfer reactions (Section **V,15**). In addition we have broadened the range of the preparative reactions included, particularly by the expansion of the sections on alicyclic compounds, heterocyclic compounds, amino acids and carbohydrates. A total of about 120 new experiments has been introduced, all of which have been checked in these laboratories. A literature reference is given for modifications or extensions to existing methods or techniques which we have not ourselves checked experimentally.

All the experiments which have been retained have been carefully scrutinised and any errors or omissions which were apparent, or known to us, have been corrected. Throughout, quantities of reagents and reactants have been expressed in molar amounts and yields calculated as a percentage of theory. The theoretical discussions and mechanistic interpretations which formed the introductions to major sections in the preparative chapters of the previous edi-

tion have been completely rewritten. In revising and up-dating the nomenclature we have been guided by the IUPAC rules for the Nomenclature of Organic Compounds, particularly with regard to the retention of established trivial names.

Every laboratory worker, and particularly those in supervisory roles, should be fully aware of the hazards associated with particular compounds and procedures, and should ensure that safe working habits are adopted. We have endeavoured to point out in the text any hazards associated with specific experiments; a comprehensive general introduction to safety is to be found in Section **1,3**. This, together with the cross-referencing provided by the indexes, should enable the dangers associated with the use of any particular material or operation to be ascertained.

The chapter on Qualitative Organic Analysis (Chapter VII) has been rewritten and now includes all the reactions for the characterisation of functional groups and the preparation of derivatives previously scattered throughout the book. This chapter also includes substantial new Sections on the interpretation of infrared, ultraviolet–visible nuclear magnetic resonance, and mass spectra of organic compounds together with a range of illustrative spectra. The Tables of Physical Constants previously dispersed throughout the book have been gathered together in Chapter VIII and Spectroscopic Correlation Tables are included in Appendices 2–4. Appendix 1, The Literature of Organic Chemistry, surveys the range of journals, monographs, texts, compilations of data, etc., which are available to the organic chemist.

We wish to thank Dr G. H. Jeffery, C.Chem., F.R.I.C., formerly Acting Head of Department who was instrumental in initiating our involvement in this project, and to Dr T. C. Downie, C.Chem., F.R.I.C. and Dr B. R. Currell, C.Chem., F.R.I.C., successive Heads of School of Chemistry in this Polytechnic for allowing the use of laboratory facilities during the work of revision. We also wish to express our thanks to Mr V. Kyte who was responsible for many of the diagrams of the previous edition, and willingly undertook the complete redrawing of retained and new material to the total of some 180 illustrations. The considerable work involved in typing from the manuscript and retyping was shared by Mrs V. Rogers and the late Mrs G. E. Tatchell; our thanks are due to both for their patience and perseverance which was such as to lighten the work involved in this extensive revision.

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September 1976

PREFACE TO FIRST EDITION

THE present volume is an attempt to give to students of practical organic chemistry the benefit of some twenty years' experience in research and teaching of the subject. The real foundations of the author's knowledge of the subject were laid in 1925–1929 when, as a research student at the Imperial College under the late Professor J. F. Thorpe, F.R.S., he was introduced to the methods and experimental technique employed in a large and flourishing school of research in organic chemistry. Since that period the author and his students have been engaged *inter alia* in researches on *Physical Properties and Chemical Constitution* (published in the Journal of the Chemical Society) and this has involved the preparation of over a thousand pure compounds of very varied type. Many of the new procedures and much of the specialised technique developed and employed in these researches are incorporated in this book. Furthermore, new experiments for the elementary student have emanated from these researches; these have been tried out with large classes of undergraduate students over several sessions with gratifying success and have now been included in the present textbook.

In compiling this book, the author has drawn freely from all sources of information available to him—research notes, original memoirs in scientific journals, reference works on organic chemistry, the numerous textbooks on practical organic chemistry, and pamphlets of manufacturers of specialised apparatus. Whilst individual acknowledgement cannot obviously be made—in many cases the original source has been lost track of—it is a duty and a pleasure to place on record the debt the writer owes to all these sources. Mention must, however, be made of *Organic Syntheses*, to which the reader is referred for further details of many of the preparations described in the text.

The book opens with a chapter on the theory underlying the technique of the chief operations of practical organic chemistry: it is considered that a proper understanding of these operations cannot be achieved without a knowledge of the appropriate theoretical principles. Chapter II is devoted to a detailed discussion of experimental technique; the inclusion of this subject in one chapter leads to economy of space, particularly in the description of advanced preparations. It is not expected that the student will employ even the major proportion of the operations described, but a knowledge of their existence is thought desirable for the advanced student so that he may apply them when occasion demands.

Chapters III and IV are confined to the preparation and properties of

Aliphatic Compounds and Aromatic Compounds respectively. This division, although perhaps artificial, falls into line with the treatment in many of the existing theoretical textbooks and also with the author's own lecture courses. A short theoretical introduction precedes the detailed preparations of the various classes of organic compounds: it is recommended that these be read concurrently with the student's lecture course and, it is hoped, that with such reading the subject will become alive and possess real meaning. The partition of the chapters in this manner provides the opportunity of introducing the reactions and the methods of characterisation of the various classes of organic compounds; the foundations of qualitative organic analysis are thus laid gradually, but many teachers may prefer to postpone the study of this subject until a representative number of elementary preparations has been carried out by the student. The division into sections will facilitate the introduction of any scheme of instruction which the teacher considers desirable.

Chapters V–X deal respectively with Heterocyclic and Alicyclic Compounds; Miscellaneous Reactions; Organic Reagents in Inorganic and Organic Chemistry; Dyestuffs, Indicators and Related Compounds; Some Physiologically-Active Compounds; and Synthetic Polymers. Many of these preparations are of course intended for advanced students, but a mere perusal of the experimental details of selected preparations by those whose time for experimental work is limited may assist to impress them on the memory. Attention is particularly directed to the chapter on Organic Reagents in Inorganic and Organic Chemistry. It is always a good plan to set advanced students or adequately-trained laboratory assistants on the preparation of those compounds which are required in the laboratory for organic and inorganic analysis; the resulting cost is comparatively low (for *o*-phenanthroline, for example, it is less than one-tenth of the commercial price) and will serve to promote the use of these, otherwise relatively expensive, organic reagents in the laboratory.

Chapter XI is devoted to Qualitative Organic Analysis. The subject is discussed in moderate detail and this, coupled with the various Sections and Tables of Physical Constants of Organic Compounds and their Derivatives in Chapters III and IV, will provide a satisfactory course of study in this important branch of chemistry. No attempt has been made to deal with Quantitative Organic Analysis in this volume.

The textbook is intended to meet the requirements of the student of chemistry throughout the whole of his training. Considerable detail is given in those sections of particular interest to the elementary student; in the author's opinion it is the duty of a writer of a practical textbook to lay a secure foundation of sound experimental technique for the beginner. The subject matter of the book is sufficiently comprehensive to permit the teacher to cover any reasonable course of instruction. It will be observed that the scale of the preparations varies considerably; the instructor can easily adapt the preparation to a smaller scale when such a step is necessary from considerations of cost and time or for other reasons. Quantities of liquid reagents are generally expressed as weights and volumes: the latter refer to a temperature of 20°. The book will be suitable for students preparing for the Pass and Honours (General and Special) B.Sc. of the Universities, the A.R.I.C. and the F.R.I.C. (Organic Chemistry). It will also provide an introduction to research methods in organic chemistry and, it is hoped, may serve as an intermediate reference book for practising organic chemists.

Attention is directed to the numerous references, particularly in Chapter II on Experimental Technique, to firms supplying specialised apparatus. The author has usually had first-hand experience with this apparatus and he feels that some readers may wish to know the present source of supply and also from whom to obtain additional information. It must be mentioned that most of the specialised apparatus has been introduced to the market for the first time by the respective firms after much development research and exhaustive tests in their laboratories. A reference to such a firm is, in the writer's opinion, equivalent to an original literature reference or to a book. During the last decade or two much development work has been carried out in the laboratories of the manufacturers of chemical apparatus (and also of industrial chemicals) and some acknowledgement of the great help rendered to practical organic chemists by these industrial organisations is long overdue; it is certainly no exaggeration to state that they have materially assisted the advancement of the science. A short list of the various firms is given on the next page.

ARTHUR I. VOGEL.
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