

EXPERIMENTAL ORGANIC CHEMISTRY

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PREFACE

This book acquaints students with all basic laboratory procedures, presented in four major parts: (I) Techniques, (II) Experiments, (III) Organic Qualitative Analysis, and Appendixes.

TECHNIQUES

The 20-chapter Techniques section acquaints the student with all common organic techniques, and has a relatively simple illustrative experiment with each. Students will occasionally find underlined plays on words which refer to some part of the subject matter at hand. This is not only a matter of interest and perhaps humor, but one of pedagogy as well. It helps train the research-oriented student to look for relationships that are not always immediately apparent.

EXPERIMENTS

The Experiments section introduces more complex experiments than those in the Techniques section. The experiments commonly involve synthetic procedures followed by workup and analysis requiring more than one technique. The experiments are arranged largely by compound class, but there are also sections on famous name reactions, redox, kinetics, acidity and neutralization, and polymers. There are 50 major experiments in addition to those in the Techniques and Organic Qualitative Analysis sections. Basically, each experiment is in the following format: introduction, theory and discussion, techniques, experimental part, writing the discussion, prelaboratory and postlaboratory exercises.

ORGANIC QUALITATIVE ANALYSIS

This part of the book encompasses most of the wet analytical techniques that are required for analysis of major functional groups. It can be used as a mini course on organic wet analysis for characterization and/or identification of a variety of tabulated unknowns, or it can be used as a tool to help identify products obtained in the Techniques and Experiments sections.

The section on qualitative organic analysis has been keyed to each experiment so that the instructor can easily assign wet analytical methods as an adjunct to the experiment.

APPENDIXES

There are three appendixes. Appendix A includes the less-referred-to techniques: sub-limation, density determination, and molecular weight determinations. Appendix B is a pronunciation guide, and Appendix C presents a compilation of chemical hazards.

GENERAL APPROACH

Presentations of theory and technique have been presented *separately* so that students do not have to search through theoretical discussions in order to find details of techniques. Each experiment presents enough theory and technique so that students are able to *fully comprehend* reaction and procedure. Attempt has been made to write procedures with such care that the instructions are complete and easy to follow—easy enough so that a good share of prelaboratory lectures are obviated, thereby saving precious laboratory time and curtailing the many procedural questions students often have. A set of prelaboratory exercises accompanying each experiment help the students to carefully consider what they are going to do in the laboratory before beginning work. The writer has been using prelaboratory exercises for about 10 years and has found this approach extremely helpful in promoting understanding and recall. It also saves laboratory time by requiring the students' prior organization of thought process.

Whenever topics are presented which are generally found in detail in lecture text-books, students are referred to these sources. However, the theory and discussion sections in this book emphasize electron transfer pathways and present fuller discussions of each experiment or technique than are found in many lecture textbooks and in most other laboratory textbooks. The writer believes that it is better to make possible a full understanding of seventy experiments than a half-understanding of one hundred.

Because for most students one of the most difficult areas of laboratory work involves writing a good discussion of results, each experiment includes suggestions for the same.

Students are led into more independent development of procedures by Experiment 37 (which requires that the students write their own procedures by analogy), Part XIX (which discusses synthetic sequences), and the techniques on searching the chemical literature and report writing.

NOMENCLATURE

The common practice of organic laboratory books of indiscriminately mixing common and systematic nomenclature is confusing for students and is not a sound educational technique. This book uses that nomenclature that seems to be most commonly employed, but with parenthetical references to the other method. Whenever possible, the IUPAC system has been given preference.

PRONUNCIATION GUIDE

Pronunciation is one of the most neglected areas of chemical study. Many students hesitate in or feel foolish about using words that they do not know how to pronounce. Because proper pronunciation is an important part of chemical communication, Appendix B contains an alphabetized pronunciation guide to many of the chemical and chemically related words used in this book.

SPECTROSCOPY

The IR and NMR techniques do not repeat much of the theoretical detail generally found in lecture textbooks, but enlarge on aspects of a topic which are necessary for student understanding of laboratory processes. Procedural details have been made as complete as practicable.

Computer usage is increasingly important in the chemical industry, in medicine, in colleges, and in other areas. To start acquainting students with the chemistry-computer interface, several experiments have been included which utilize computer analysis of experimental results.

MASS SPECTROMETRY

Mass spectrometry is not normally included in organic laboratory books because the equipment is too expensive. However, this book presents an introduction to mass spectrometric analysis via microcomputer simulations.

The mass spectrometry simulation permits for the first time the teaching of mass spectrometry to all students in the undergraduate laboratory. The mass spectrometry program contains 17 different unknown alcohols, aldehydes, and ketones arranged as 36 unknown samples. Some of these unknowns are programmed as GLC samples and require instrument control settings which differ from those used under standard conditions. The students must learn what control setting to use in order to obtain meaningful spectra. They can adjust the controls to maximize or minimize the size of the molecular ion (M) peaks or the (M+1) peaks. The writer has tried to make the results of the simulation as realistic as possible: The sizes of the peaks actually change relative to the control panel settings, and at high ionizing currents, one observes small peaks due to pump oil. Very complete information about unknowns is provided in the Instructor's Manual, including mechanisms of fragmentations.

LABORATORY NOTE-KEEPING

The Laboratory Notebook Technique is the most comprehensive to be found in any organic laboratory book. If the instructor chooses to follow through with the most rigorous procedures discussed, students learn record-keeping techniques that will be acceptable to both university and chemical industry, and will stand up in controversial patent disputes. Also in this section is a review of the various calculations an organic laboratory student must make.

LABORATORY SAFETY

This book treats every experiment as an adjunct in safety, makes available in Appendix C the chemical hazards of the chemicals used in this book, and calls attention to every likely operational hazard. Particularly hazardous chemicals, like benzene, have been avoided. Safety considerations are so much more complete than those found in other organic laboratory books that this text might indeed set a new standard for the same.

CONSERVATION OF CHEMICALS

The cost of organics is an increasingly important consideration. In most cases, the laboratory instructions in this book call for one-quarter to one-half of the amounts of chemicals characteristically required by similar macroscale laboratory textbooks. This book has a built-in program and specific instructions to students for conserving materials. Solvents are recycled, and the products of many processes can be used in later experiments. The technique on process economics emphasizes the necessity of conservation and cost effectiveness.

PROCESS ECONOMICS

A cost analysis of chemical process is the subject of Technique 20. This analysis provides the student with a basis for understanding economics of the chemical industry and is accompanied by a computer program which drastically reduces the time required for analysis.

USABLE PRODUCTS

To further add interest to laboratory work, a number of experiments have been devised that lead to usable products. None of these experiments was chosen solely for its product value. Each relates to a theory and/or technique in the study of organic chemistry laboratory.

Orientation and Organization Laboratory Safety Basic Laboratory Equipment and Techniques

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