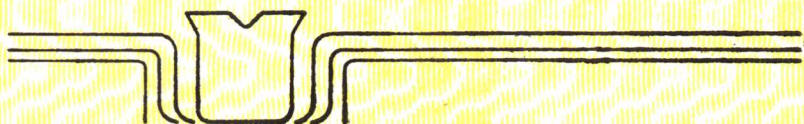


# Techniques and Experiments for Organic Chemistry

FIFTH EDITION

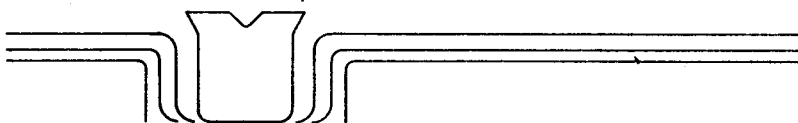


Addison Ault

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FIFTH EDITION



Addison Ault  
Cornell College

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# Preface

This book is intended for use in the laboratory part of an introductory course in organic chemistry. The overall organization of this fifth edition is the same as that of the fourth edition. Part 1 contains general descriptions of the theory and practice of the most common laboratory techniques of organic chemistry; Part 1 also presents directions for a number of exercises that illustrate these techniques. Part 2 contains experiments that range from the purification of natural products to one-step transformations to multi-step syntheses. Again, as in the fourth edition, this book includes a number of *variations*, or alternative experiments, that call for a different starting material and that require the student to provide some of the details of the experimental procedure. By using these variations, the instructor can move away from the detailed recipe, or "cookbook," approach to laboratory work.

The first sections of Part 1 contain a discussion of laboratory safety, a description of the glassware used in the organic chemistry laboratory, advice on cleaning up, directions for writing up a laboratory notebook, and an introduction to the chemical literature. After these preliminary topics come discussions of procedures for the isolation and purification of organic substances and of techniques such as crystallization, distillation, extraction, and the chromatographic methods. These are followed by sections on physical methods for the identification and characterization of organic compounds. These methods include the determination of boiling point and melting point, the determination of properties such as density, index of refraction, and optical rotation, and the recording and interpretation of infrared and nuclear magnetic resonance spectra. Next come chemical methods of identification and characterization: qualitative tests for the elements, qualitative tests for functional groups, and procedures for the preparation of derivatives. (The physical properties of selected unknowns and the melting points of their derivatives are again listed in tables in the Appendix.) Finally, in Part 1, there are

sections that describe the apparatus and techniques used in the laboratory operations of organic chemistry.

Part 2 first presents experiments that exemplify the separation and purification of substances, experiments such as the isolation of cholesterol from gallstones and of lactose from powdered milk, the recovery of (R)-(+)-limonene from grapefruit or orange peel, the isolation of the two enantiomeric forms of carvone (these enantiomers have different odors), and the resolution of  $\alpha$ -phenylethylamine. Part 2 then continues with a variety of one-step transformations that illustrate the chemistry of the functional groups and concludes with a set of multi-step syntheses: the preparation of  $\Delta^4$ -cholestene-3-one from cholesterol, the syntheses of tetraphenylcyclopentadienone, sulfanilamide, *p*-phenetidine, and 1-bromo-3-chloro-5-iodobenzene, and the preparation of a merocyanine dye.

The fifth edition retains all of the experiments of the fourth edition, and several new experiments have been added. These include the isolation of piperine from black pepper, the borohydride reduction of vanillin to vanillyl alcohol, the preparation of the analgesic *p*-ethoxyacetanilide from *p*-aminophenol, the synthesis of "coconut aldehyde," and a bootstrap synthesis—the preparation of two moles of *p*-phenetidine from one mole of *p*-phenetidine.

I believe that students find lab work more interesting when they understand why procedures work and can participate in the planning of experiments. I therefore emphasize explanations and provide for student participation in the planning of experiments by allowing students to choose one of several similar preparations of the same compound or to adapt a procedure for the preparation of one compound to the preparation of another, as in the variations.

Finally, I am happy to acknowledge the interest and other contributions of Dr. David Todd (The Worcester Polytechnic Institute, Massachusetts) and Dr. Jeffrey Keiser (Coe College, Iowa), the editorial and production assistance of Allyn and Bacon, Inc., and the continuing interest and encouragement of my wife, Janet, and of my children, Margaret, Warren, Tad, Peter, and Emily.

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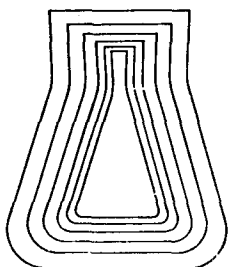
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# 1 Laboratory Operations

- Preliminary Topics
- Separation of Substances; Purification of Substances
- Determination of Physical Properties
- Determination of Chemical Properties; Qualitative Organic Analysis
- Apparatus and Techniques for Chemical Reactions

Part 1 of this book describes many of the practical operations that one might perform in the organic chemistry laboratory. The first section in this part presents a discussion of laboratory safety, and this is followed by sections that describe the glassware used in the organic laboratory, the laboratory notebook, the literature of organic chemistry, and techniques for cleaning up. The remaining sections are presented in groups: (1) methods of separation and purification, (2) methods for the determination of physical properties, (3) methods for the determination of chemical properties, and (4) the apparatus and techniques for carrying out chemical reactions.

The sections on the theory and techniques of separation and purification include discussions of filtration, re-

## 2 LABORATORY OPERATIONS

crystallization, distillation, steam distillation, sublimation, extraction, chromatography, and removal of water.

The sections on the determination of physical properties and the dependence of physical properties on molecular structure include boiling point, melting point, density, index of refraction, optical rotation, molecular weight, and solubility characteristics, as well as the more recently developed spectrometric methods: infrared, ultraviolet-visible, nuclear magnetic resonance, and mass spectrometry.

The sections concerning the determination of chemical properties include qualitative tests for elements, qualitative tests for functional groups, and reactions for the formation of derivatives.

The sections on apparatus and techniques for chemical reactions include methods for heating and cooling, for stirring, for adding reagents, and for working up the reaction and isolating the product.