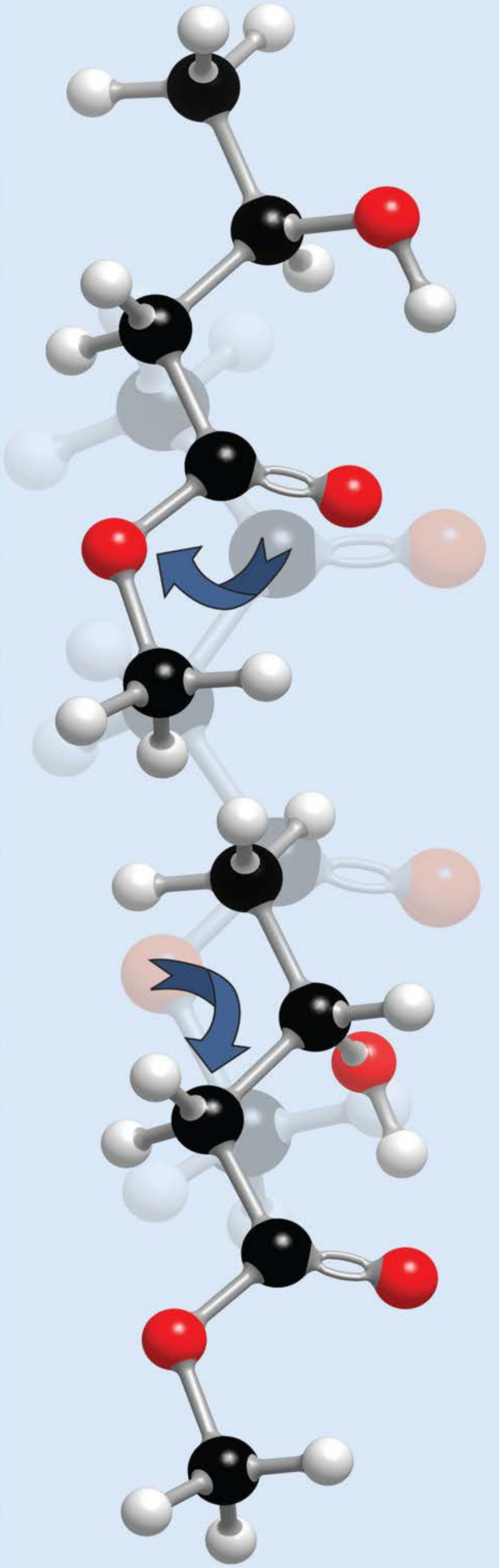
CENGAGE LEARNING LABORATORY SERIES for
Organic Chemistry

Experimental Organic Chemistry

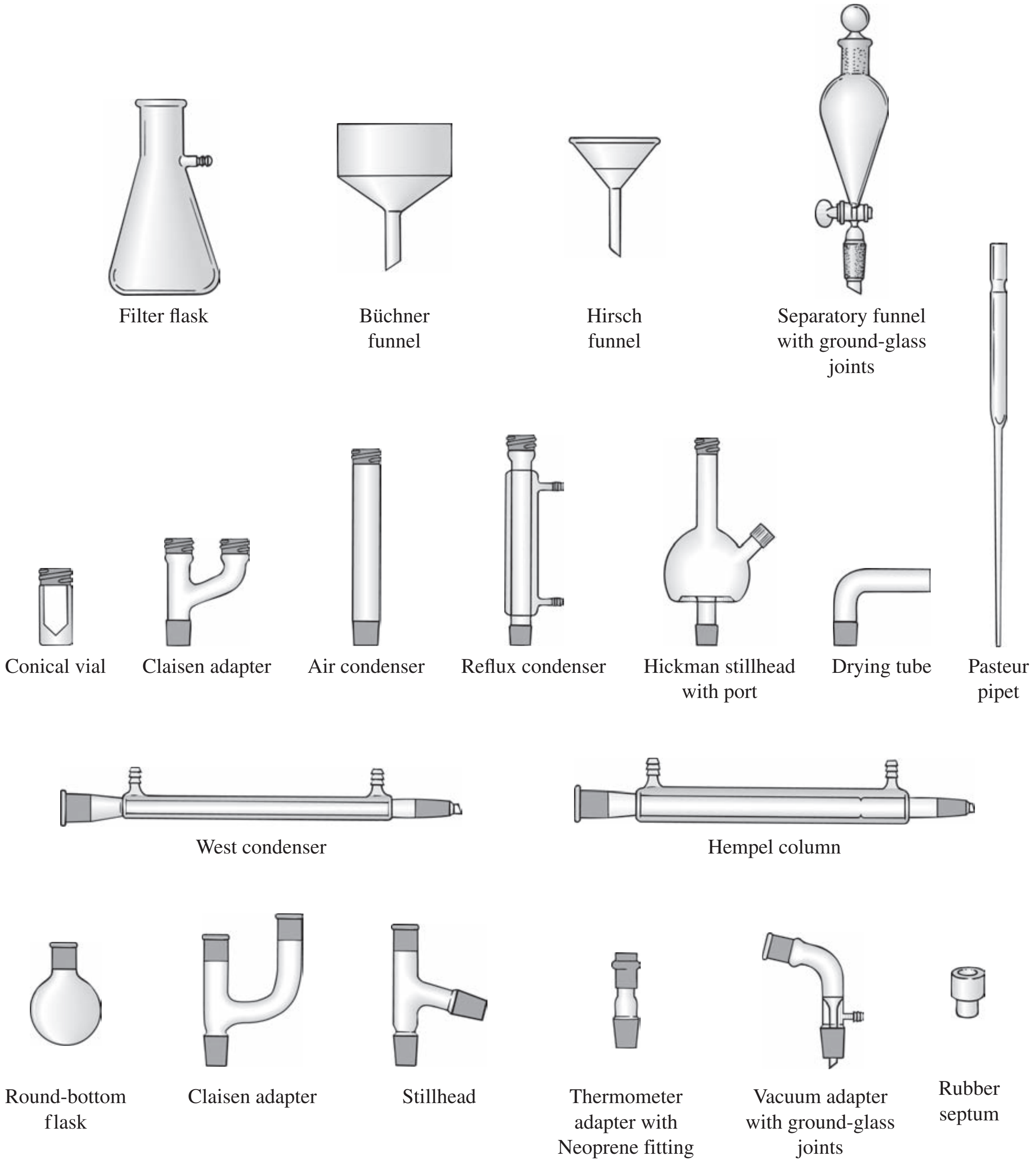
A Miniscale and Microscale Approach

SIXTH EDITION



John C. Gilbert
Stephen F. Martin

Equipment Commonly Used in the Organic Chemistry Laboratory



First Aid in Case of an Accident

The occurrence of an accident of any kind in the laboratory should be reported promptly to your instructor, even if it seems relatively minor.

FIRE

Your first consideration is to remove yourself from any danger, *not* to extinguish the fire. *If it is possible to do so without endangering yourself*, turn off any burners and remove containers of flammable solvents from the immediate area to prevent the fire from spreading. For the most effective use of a fire extinguisher, direct its nozzle toward the *base* of the flames. Burning oil may be put out with an extinguisher classified for use on "ABC" type fires.

If your clothing is on fire, DO NOT RUN; rapid movement will only fan the flames. Roll on the floor to smother the fire and to help keep the flames away from your head. Your neighbors can help to extinguish the flames by using fire blankets, laboratory coats, or other items that are immediately available. Do not hestitate to aid your neighbor if he or she is involved in such an emergency; a few seconds delay may result in serious injury. A laboratory shower, *if close by*, can be used to extinguish burning clothing, as can a carbon dioxide extinguisher, which must be used with care until the flames are extinguished *and only if the flames are not near the head*.

If burns are minor, apply a burn ointment. In the case of serious burns, do not apply any ointment; seek professional medical treatment at once.

CHEMICAL BURNS

Areas of the skin with which corrosive chemicals have come in contact should be immediately and thoroughly washed with soap and warm water. If the burns are minor, apply burn ointment; for treatment of more serious burns, see a physician.

Bromine burns can be particularly serious. Such burns should first be washed with soap and warm water and then thoroughly soaked with 0.6 *M* sodium thiosulfate solution for 3 h. Apply cod liver oil ointment and a dressing; see a physician.

If chemicals, in particular corrosive or hot reagents, come in contact with the eyes, immediately *flood* the eyes with water *from the nearest outlet*. It is best to use a specially designed eyewash fountain if one is available in the laboratory. *Do not touch the eye*. The eyelid as well as the eyeball should be washed with water for several minutes. In all instances where sensitive eye tissue is exposed to corrosive chemicals, an ophthalomologist should be consulted as soon as possible.

CUTS

Minor cuts may be treated by ordinary first-aid procedures; seek professional medical attention for serious cuts. If severe bleeding indicates that an artery has been severed, attempt to stop the bleeding with compresses and pressure; a tourniquet should be applied only by those who have received first-aid training. Arrange for emergency room treatment at once.

A person who is injured severely enough to require a physician's treatment *should be accompanied* to the doctor's office, or infirmary, even if he or she claims to be all right. Persons in shock, particularly after suffering burns, are often more seriously injured than they appear to be.

List of New Experiments

Ch 10 C. Bromination of (*E*)-Cinnamic Acid

Ch 17 B. Transfer Hydrogenation of Cinnamic Acid Derivatives
A. Tartaric Acid-Mediated Enantioselective Reduction of Methyl Acetoacetate

Ch 19 Preparation of 3-Ethylhex-5-En-3-Ol
Preparation of 4'-Methyl-(1,1'-Biphenyl)-4-Methanol

List of New Discovery Experiments

Ch 3 C. Who Else Has My Compound?

Ch 10 C. Bromination of (*E*)-Cinnamic Acid

Ch 14 Competing Nucleophiles in S_N Reactions
Competition between Substitution and Elimination

Ch 15 B. Electrophilic Aromatic Bromination of Monosubstituted Arenes
C. Nitration of Monosubstituted Arenes
Azo Dyes and the Chemistry of Dyeing Fabrics

Ch 17 B. Transfer Hydrogenation of Cinnamic Acid Derivatives
A. Tartaric Acid-Mediated Enantioselective Reduction of Methyl Acetoacetate

Ch 20 Identifying Unknown Esters Produced by Fischer Esterification

List of New Green Experiments

Ch 15 Friedel-Crafts Acylation of Anisole

Ch 16 C. Aerobic Oxidation of Benzylic Alcohols

Ch 18 Synthesis of Ethyl 6-Methyl-2-Oxo-4-Phenyl-1,2,3,4-Tetrahydropyrimidine-5-Carboxylate

Experimental Organic Chemistry

A Miniscale and Microscale Approach

SIXTH EDITION

John C. Gilbert

Santa Clara University

Stephen F. Martin

University of Texas at Austin

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CENGAGE Learning

***Experimental Organic Chemistry: A Miniscale and Microscale Approach*, Sixth Edition**
John C. Gilbert and Stephen F. Martin

Product Director: Mary Finch

Product Manager: Krista Mastroianni

Content Developer: Thomas Martin

Managing Developer: Peter McGahey

Product Assistant: Morgan Carney

Associate Media Developer: Elizabeth Woods

Senior Marketing Manager: Julie Schuster

Art and Cover Direction, Production Management, and Composition: Lumina Datamatics, Inc

Manufacturing Planner: Judy Inouye

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Cengage Learning
20 Channel Center Street
Boston, MA 02210
USA

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Contents in Brief

Table of Contents

Preface

The management and teaching of an introductory laboratory course in organic chemistry is ever-changing, even though the fundamental chemical principles remain the same. Some of the compelling reasons for innovation and change are linked to the increasing cost associated with purchase and disposal of the chemicals used. There is the added concern of their possible toxicological hazards, both to students and to the environment. These factors dictate that many experiments be performed on reduced scales according to procedures commonly termed as *miniscale* (sometimes called small-scale) and *microscale*. This edition of our textbook maintains our practice of providing both miniscale and microscale procedures for most experiments. This unusual feature gives instructors maximal flexibility in customizing the course for use of apparatus and glassware already on hand and to suit the specific needs of you, the student.

The experiments are thoughtfully selected to introduce you to the common laboratory practices and techniques of organic chemistry and to illustrate the chemistry of the wide range of functional groups that are present in organic molecules. Some experiments are designed to familiarize you with the kinetic and thermodynamic principles underlying chemical reactions. Others allow you to synthesize specific compounds—some of which are found in nature or are of commercial importance—using reactions that are fundamental to organic synthesis. Still others introduce you to *discovery-based* and *green-chemistry* approaches. The discovery-based procedures—there are more than 50 of these in this edition—allow you to develop your own protocols for addressing a particular question experimentally, as you might do in a research laboratory. Discovery experiments are listed inside the front cover and are indicated when they appear in the book with the magnifying glass icon shown in the margin. The five procedures involving green chemistry show you how some chemical transformations may be performed using more environmentally friendly procedures. Green chemistry experiments are indicated when they appear in the book with the leaf icon shown in the margin. Most of the chapters are accompanied by a Historical Highlight, which are only available online. These 21 essays focus on interesting topics in organic chemistry that we believe will broaden your interest in the subject. Overall, our hope is that your experiences in this course will inspire you to take additional laboratory and lecture courses in chemistry, to seize the opportunity to work in a research laboratory as an undergraduate student, and perhaps even to pursue a career in research.

Background Information

Our textbook is distinct from many other laboratory manuals because the focused discussions preceding each Experimental Procedure provide the essential theoretical and "how-to" background, so other sources need not be consulted in order to understand the mechanistic and practical aspects of the specific reactions and procedures being performed. These discussions offer the advantage of making the textbook self-contained, and because they focus on the experiments themselves, they also significantly augment the material found in your lecture textbook.

Experimental Procedures

The *miniscale approach* appeals to instructors who believe in the importance of performing experiments on a scale that allows isolation and characterization of products using conventional laboratory glassware. The quantities of starting materials used are usually in the range of 1–3 g, so the costs associated with purchasing and disposing of the chemicals are modest. The amounts of material may be easily handled, and it is possible to develop the techniques required to purify the products and characterize them by comparing their physical properties with those reported in the scientific literature. You will also be able to characterize the starting materials and products by spectroscopic techniques, so that you can see how their spectral properties differ. In short, you will be able to experience the real world of organic chemistry in which usable quantities of compounds are synthesized.

The *microscale approach* is especially attractive for minimizing the cost of purchasing and disposing of chemicals. The specialized glassware and other apparatus required for performing experiments on such small scales is now readily available. Indeed, many of the components found in a microscale kit are also found in the advanced organic laboratory, where trained researchers often work with minute amounts of material. The amounts of starting materials that are used in these procedures are often only 100–300 mg. Because of the small quantities of materials being handled, you must be meticulous in order to isolate products from microscale reactions. Purifying small quantities of materials by distillation or recrystallization is often tedious, so it will frequently be impractical to characterize pure products. Nevertheless, the experiments performed on the microscale should provide tangible quantities of material so that you can verify that the product was formed using chemical tests as well as some spectroscopic and analytical techniques.

Organization

The experiments we have included are intended to reinforce concepts given in the lecture course in organic chemistry and to familiarize you with the techniques that modern organic chemists routinely use. The basic types of apparatus you will need are described in Chapter 2. In addition, videos illustrating the steps required to assemble many of the set-ups are available online, and we urge you to view these *prior* to going to the laboratory. In subsequent chapters, we provide figures in the margins of the pages to remind you how the assembled apparatus appears. The procedures in Chapters 3–6 are designed to introduce you to the different techniques for distillation, liquid-liquid and liquid-solid extraction, and thin-layer, column, and gas-liquid chromatography; the basic principles for these techniques are also described in their respective chapters. The spectroscopic methods that are fundamental to analyzing organic compounds are described in Chapter 8. Experiments that illustrate concepts such as selectivity of free-radical substitution (Chapter 9), kinetic and thermodynamic control of reactions (Chapter 13), kinetics of nucleophilic substitution reactions (Chapter 14), kinetics and regiochemistry of electrophilic aromatic substitution reactions (Chapter 15), and the stereochemistry and regiochemistry of addition reactions (Chapters 10, 11, 12, and 17) are intended to provide a better understanding of these important subjects. Other experiments illustrate specific chemical transformations such as the generation, reactions, and rearrangements of carbocations (Chapters 10 and 15), electrophilic aromatic and nucleophilic substitution processes (Chapters 15 and 14, respectively), eliminations (Chapters 10 and 11), oxidations and reductions (Chapters 16 and 17, respectively), nucleophilic additions to carbonyl compounds and imines (Chapters 17 and 18, respectively), the generation and reactions of Grignard and organozinc reagents (Chapter 19), and the formation of various carboxylic acid derivatives (Chapter 20). An experiment in Chapter 15 will introduce you to the fun of making your own dyes, and in Chapter 20 you will be able to observe the fascinating phenomenon of chemiluminescence. The value of enzymes for effecting enantioselective reactions is illustrated in Chapter 17. Because the current practice of organic chemistry in industry frequently involves multistep transformations, several examples of multi step synthesis are contained in Chapter 21. Many industrial processes rely on the use of transition-metal catalysts, and you will be able to study one such reaction in Chapter 19. Experiments designed to introduce you to basic concepts of carbohydrate chemistry and polymer chemistry are provided in Chapters 22 and 23, respectively, and the experiments given in Chapter 24 give you an opportunity to explore one aspect of the world of bio-organic chemistry through synthesis of a dipeptide. A rational approach to solving the structures of unknown compounds with and without the aid of spectroscopic data is given in Chapter 25, which is available online.

Spectroscopic Techniques

Spectroscopy may be the single most powerful tool for analyzing organic compounds. Consequently, thorough discussions of the theory and practical techniques for infrared, nuclear magnetic resonance (including ^{1}H and ^{13}C NMR), UV-Vis, and mass spectrometry are presented in Chapter 8. To reinforce the basic spectroscopic principles and to provide an opportunity for interpreting spectroscopic data, the infrared and nuclear magnetic spectra of all of the organic starting materials and products are provided in this textbook and online. It is also possible for you to perform simple manipulations of the ^{1}H NMR and IR spectra that are available at the website. For example, you will be able to measure chemical shifts, integrals, and coupling constants directly on the ^{1}H NMR spectra. You will also be able to determine the position of an absorption in the IR spectrum that is associated with a specific functional group. This "hands-on" experience has proved an invaluable aid in teaching the basics of interpreting ^{1}H NMR and IR spectra and is unique to this laboratory textbook.

Safety and the Environment

Important sections entitled "Safety Alert" and "Wrapping It Up" are included with each experimental procedure. The information in the "Safety Alert" is designed to inform you and your instructor of possible hazards associated with the operations being performed. The abbreviated Material Safety Data Sheets (MSDSs) are available online and provide additional information regarding flammability and toxicological properties of the chemicals being used and produced. Because of the flammable nature of the solvents and the chemicals that are handled in the laboratory, the use of *flameless* heating is emphasized and should be implemented in order to make the laboratory a safe workplace. The guidelines and methods in the "Wrapping It Up" section will familiarize you with the proper procedures for disposing of chemicals and other by-products after you have completed the experiment. Using these recommended methods will help protect the environment and lessen the costs associated with the ultimate disposal of these materials.

Essays

A feature of most of the chapters in the textbook is a Historical Highlight which are only available online. These 21 essays, some of which are biographical in nature, are designed to familiarize you with the lives of some of the chemical pioneers who have advanced science. These accounts will also provide you with a sense of the excitement and insights of individuals whose scientific observations form the basis for some of the experiments you will perform. Other essays are intended to relate organic chemistry to your everyday life. We hope they will whet your appetite for the subject of organic chemistry and enrich your experience as you further develop your scientific expertise.

Pre-Lab and Post-Lab Exercises

Each experiment is accompanied by two sets of questions. The Pre-Lab Exercises are provided online and are designed to test your understanding of potential safety hazards and basic concepts, so you will be able to perform the experiments and techniques safely and successfully. Because these questions will assist you in preparing for work in the laboratory, we strongly recommend that you answer them *before* performing the experiment. The Post-Lab Exercises are found under the heading "Exercises" after each Experimental Procedure. These questions are written to reinforce the principles that are illustrated by the experiments and to determine whether you understand the observations you have made and the operations you have performed. Furthermore, questions on spectroscopy will help you develop the skills required to interpret IR and ^{1}H and ^{13}C NMR spectra.

Significant Changes from the Fifth Edition

This edition of the textbook includes a total of 15 new experiments. Ten of these are discovery experiments in which you will have an opportunity to identify unknown compounds and to explore reactivity and mechanism. Three new green chemistry experiments have been added that will enable you to learn how chemical reactions can be performed in an environmentally friendly manner. Finally, four experiments have been added so you can explore several modern methods to induce selective transformations.

Chapter 25—*Identifying Organic Compounds* and Chapter 26—*The Literature of Organic Chemistry* and the Historical Highlights are only available online. Moving these documents online provided space to add new experiments in various chapters as needed.

This edition includes a new OWLv2 LabSkills program that can be bundled with the textbook. LabSkills provides students the opportunity to practice skills and prepare for real laboratory classes in a safe environment. Students work through the assignments in advance of the lab so they can actively rehearse common practical techniques by engaging with simulations, videos, and quizzes. Because LabSkills focuses on techniques, instructors can customize assignments to align with the experiments that students will perform in any given lab session. The online course will also offer assessment specific to this edition's experiments, and include a MindTap Reader. The new OWLv2 course with MindTapReader includes assignable prelab exercises integrated with LabSkills Safety and Techniques videos. All content is mobile compatible. The eBook contains interactive spectra.

Feedback

As always, we seek your comments, criticisms, and suggestions for improving our textbook. Despite our best efforts, we are certain that there are typographical errors and the like that have escaped our notice, and we would appreciate your bringing them to our attention; our e-mail and snail-mail addresses are provided below. No matter how busy we might be, we shall respond to any messages you send.

Supporting Materials

Please visit http://www.cengage.com/chemistry/gilbert/eoc6e for information about student and instructor resources for this book and about custom versions.

Acknowledgments

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John C. Gilbert
jgilbert@scu.edu
Department of Chemistry & Biochemistry
Santa Clara University
Santa Clara, CA 95053

Stephen F. Martin
sfmartin@mail.utexas.edu
Department of Chemistry
The University of Texas at Austin
Austin, TX 78712

Introduction, Record Keeping, and Laboratory Safety

This chapter sets the stage as you undertake the adventure of experimental organic chemistry. Although we may be biased, we think that this laboratory experience is one of the most valuable you will have as an undergraduate student. There is much to be learned as you progress from the relatively structured format of your first laboratory course in organic chemistry to the much less defined experimental protocols of a scientific research environment. The laboratory practices described in the following sections should serve you well in the journey.

1.1 INTRODUCTION

The laboratory component of a course in organic chemistry has an important role in developing and augmenting your understanding of the subject matter. The theoretical concepts, functional groups, and reactions presented in the lecture part of the course may seem abstract at times, but they are more understandable as a result of the experiments you perform. The successes, challenges, and, yes, frustrations associated with the "hands-on" experience gained in the laboratory, as you gather and interpret data from a variety of reactions, provide a sense of organic chemistry that is nearly impossible to communicate in formal lectures. For example, it is one thing to be told that the addition of bromine (Br_2) across the π-bond of most alkenes is a rapid process at room temperature. It is quite another to personally observe the *immediate* decoloration of a reddish solution of bromine in dichloromethane (Br_2/CH_2Cl_2) as a few drops of it are added to cyclohexene. The principles developed in the lectures will help you to predict what reaction(s) should occur when various reagents are combined in experimental procedures and to understand the mechanistic course of the process(es). Performing reactions allows you to test and verify the principles presented in lecture. Moreover, careful and thoughtful analysis of the observations and results of the experiments will foster the skills in critical thinking that are a hallmark of successful scientists.

Of course, the laboratory experience in organic chemistry has another important function beyond reinforcing the concepts presented in lecture—to introduce you to the broad range of techniques and procedures that are important to the successful practice of experimental organic chemistry. You will learn how to handle a variety of chemicals safely and how to manipulate apparatus properly, talents that are critical to your success as a student of the chemical sciences. Along with becoming more skilled in the technical aspects of laboratory work, you should also develop a proper scientific approach to executing experiments and interpreting the results. By reading and, more importantly, *understanding* the concepts of this chapter, you will be better able to achieve these valuable goals.

1.2 PREPARING FOR THE LABORATORY

A common misconception students have about performing experiments is that it is much like cooking; that is, you merely follow the directions given—the "recipe"—and the desired product or data will result. Such students enter the laboratory expecting to follow the experimental procedure in a more or less rote manner. This unfortunate attitude can lead to inefficiencies, accidents, and minimal educational benefit and enjoyment from the laboratory experience.

To be sure, cooking is somewhat analogous to performing experiments. The successful scientist, just like a five-star chef, is a careful planner, a diligent worker, a keen observer, and, importantly, is fully prepared for failures! Experiments may not work despite your best efforts, just as a cake may fall even in the hands of a premier pastry chef.

The correct approach to being successful in the laboratory is *never* to begin any experiment until you understand its overall purpose and the reasons for each operation that you are to do. This means that you must *study*, not *just read*, the entire experiment *prior* to arriving at the laboratory. Rarely, if ever, can you complete the necessary preparation in 5 or 10 minutes, which means that you should not wait until just before the laboratory period begins to do the studying, thinking, and writing that are required. *Planning* how to spend your time in the laboratory is the key to efficient completion of the required experiments. Your performance in the laboratory will benefit enormously from proper advance work, and so will your grade!

The specific details of what you should do before coming to the laboratory will be provided by your instructor. However, to help you prepare in advance, we have developed a set of Pre-Lab Exercises for each of the experimental procedures we describe. These exercises are Web-based and are available online.

Your instructor may require you to submit answers to the Pre-Lab Exercises for approval before authorizing you to proceed with the assigned experiments. Even if you are not required to submit the exercises, though, you will find that answering them *prior* to the laboratory period will be a valuable educational tool to self-assess your understanding of the experiments to be performed.

You undoubtedly will be required to maintain a laboratory notebook, which will serve as a complete, accurate, and neat record of the experimental work that you do. Once more, your instructor will provide an outline of what specific information should appear in this notebook, but part of what is prescribed will probably necessitate advance preparation, which will further enhance your ability to complete the experiments successfully. The laboratory notebook is a *permanent record* of your accomplishments in the course, and you should take pride in the quality and completeness of its contents!

1.3 WORKING IN THE LABORATORY

You should be aware that experimental organic chemistry is *potentially* dangerous, because many of the chemicals used are toxic and/or highly flammable, and most of the procedures require the use of glassware that is easily broken. Careless handling of these chemicals and sloppy assembly of apparatus are sources of danger not only to you but also to those working near you. You *should* not be afraid of the chemicals and equipment that you will be using, but you should treat them with the respect and care associated with safe experimental practices. To facilitate this, there is an emphasis on the proper handling of chemicals and apparatus throughout the textbook, and the importance of paying particular attention to these subjects *cannot* be overemphasized. In a sense, laboratory safety is analogous to a chain that is only as strong as its weakest link: The possibility that an accident will occur is only as great as the extent to which unsafe practices are followed. In other words, if you and your labmates adhere to proper laboratory procedures, the risk of an accident will be minimized.

It is important that you follow the experimental procedures in this textbook closely. There is a good reason why each operation should be performed as it is described, although that reason may not be immediately obvious to you. Just as it is risky for a novice chef to be overly innovative when following a recipe, it is *dangerous* for a beginning experimentalist to be "creative" when it comes to modifying the protocol that we've specified. As you gain experience in the organic laboratory, you may wish to develop alternative procedures for performing a reaction or purifying a desired product, but *always* check with your instructor *before* trying any modifications.

Note that rather detailed experimental procedures are given early in the textbook, whereas somewhat less detailed instructions are provided later on. This is because many of the basic laboratory operations will have become familiar to you in time and need not be spelled out explicitly. It is hoped that this approach to the design of procedures will decrease your tendency to think that you are essentially following a recipe in a cookbook. Moreover, many of the experimental procedures given in the literature of organic chemistry are relatively brief and require the chemist to "fill in the blanks," so it is valuable to gain some initial experience in figuring out some details on your own.

Most of your previous experience in a chemistry laboratory has probably required that you measure quantities precisely, using analytical balances, burets, pipets, and other precise measuring devices (Secs. 2.5 and 2.6). Indeed, if you have done quantitative inorganic analysis, you know that it is often necessary to measure weights to the third or fourth decimal place and volumes to at least the first. Experiments in organic chemistry that are performed at the **microscale** level, that is, experiments in which less than about 1 mL of the principal reagents is used and the amounts of solvents are less than 2 or 3 mL, also require relatively precise measuring of quantities. For example, if you are to use 0.1 g of a reagent and your measuring device only allows measuring to the nearest 0.1 g, you could easily have as much as about 0.15 g or as little as 0.05 g of the reagent. Such deviations from the desired quantity represent significant *percentage* errors in measurement and can result in serious errors in the proportions of reagents involved in the reaction. Consequently, weights should be accurate to within about 0.01 g and volumes to within about 0.1 mL. This requires the use of appropriate analytical balances and graduated pipets.

Experiments being performed at the **miniscale** level, which we define as involving 1–5 g of reagents and usually less than about 25 mL of solvent, normally do not require such precise measuring. Weighing reagents to the nearest tenth of a gram is usually satisfactory, as is measuring out liquids in graduated cylinders, which are accurate to $\pm$10%. For example, if you are directed to use 20 mL of diethyl ether as solvent for a reaction, the volume need *not* be 20.0 mL. In fact, it probably will make little difference to the success of the reaction whether anywhere from

15 mL to 25 mL of the solvent is added. This is not to say that care need not be exercised in measuring out the amounts of materials that you use. Rather, it means that valuable time need not be invested in making these measurements highly precise.

We've inserted markers in the form of **stars** (★) in many of the experimental procedures in this textbook. These indicate places where the procedure can be interrupted without affecting the final outcome of the experiment. These markers are designed to help you make the most efficient use of your time in the laboratory. For example, you may be able to start a procedure at a point in the period when there is insufficient time to complete it but enough time to be able to work through to the location of a star; you can then safely store the reaction mixture and finish the sequence during the next laboratory period. We've *not* inserted stars at every possible stopping point but only at those where it is not necessarily obvious that interruption of the procedure will have no effect on the experimental results. Consult your instructor if in doubt about whether a proper stopping point has been reached.

As noted above, a *carefully* written **notebook** and *proper* **safety procedures** are important components of an experimental laboratory course. These aspects are discussed further in the following two sections.

1.4 THE LABORATORY NOTEBOOK

One of the most important characteristics of successful scientists is the habit of keeping a complete and understandable record of the experimental work that has been done. Did a precipitate form? Was there a color change during the course of the reaction? At what temperature was the reaction performed, and for how long did the reaction proceed? Was the reaction mixture homogeneous or heterogeneous? On what date(s) was the work performed? These are observations and data that may seem insignificant at the time but may later prove critical to the interpretation of an experimental result or to the ability of another person to reproduce your work. All of them belong in a properly kept laboratory notebook. We make suggestions for such a document in the following two sections. Your instructor may specify other items to be included, but the list we give is representative of a good notebook.

1.5 GENERAL PROTOCOL FOR THE LABORATORY NOTEBOOK

1. Use a *bound* notebook for your permanent laboratory record to minimize the possibility that pages will be lost. If a number has not been printed on each page, do so manually. Some laboratory notebooks are designed with pairs of identically numbered pages so that a carbon copy of all entries can be made. The duplicate page can then be removed and submitted to your instructor or put in a separate place for safekeeping. Many professional scientists use this type of notebook.
2. Reserve the first page of the notebook for use as a title page, and leave several additional pages blank for a Table of Contents.
3. Use as the main criterion for what should be entered in the notebook the rule that the record should be sufficiently complete so that anyone who reads it will know exactly what you did and will be able to repeat the work in precisely the way you originally did it.

4. Record all experimental observations and data in the notebook *as they are obtained*. Include the date and, if appropriate, the *time* when you did the work. In a legal sense, the information entered into the notebook *at the time of performance* constitutes the primary record of the work, and it is important for you to follow this principle. Many patent cases have been determined on the basis of dates and times recorded in a laboratory notebook. One such example is described in the Historical Highlight *The Importance of Record Keeping,* which is available online.
5. Make all entries in ink, and *do not delete anything you have written* in the notebook. If you make a mistake, cross it out and record the correct information. Using erasers or correction fluid to modify entries in your notebook is unacceptable scientific practice!

   Do not scribble notes on odd bits of paper with the intention of recording the information in your notebook later. Such bad habits only lead to problems, since the scraps of paper are easily lost or mixed up. They are also inefficient, since transcribing the information to your notebook means that you must write it a second time. This procedure can also result in errors if you miscopy the data.

   Finally, do not trust your memory with respect to observations that you have made. When the time comes to write down the information, you may have forgotten a key observation that is critical to the success of the experiment.
6. Unless instructed to do otherwise, do not copy *detailed* experimental procedures that you have already written elsewhere in your notebook; this consumes valuable time. Rather, provide a specific reference to the source of the detailed procedure and enter a *synopsis* of the written procedure that contains enough information that (1) you need not refer to the source while performing the procedure and (2) another chemist will be able to *duplicate* what you did. For example, when performing an experiment from this textbook, give a reference to the page number on which the procedure appears, and detail any *variations* made in the procedure along with the reason(s) for doing so.
7. Start the description of each experiment on a new page titled with the name of the experiment. The recording of data and observations from several different procedures on the same page can lead to confusion, both for yourself and for others who may read your notebook. If you are unable to complete the write-up of an experiment on sequential pages, be certain to specify the page(s) on which the continuation appears.

1.6 TYPES OF ORGANIC EXPERIMENTS AND NOTEBOOK FORMATS

There are two general classes of experiments, **investigative** and **preparative,** in this textbook. Investigative experiments normally involve making observations and learning techniques that are common to laboratory work in organic chemistry but do not entail conversion of one compound into another. Some examples are solubility tests, distillation, recrystallization, and qualitative organic analysis. In contrast, preparative experiments involve interconversion of different compounds. Most of the procedures described in this textbook fall into the latter category.

The format of the laboratory notebook is usually different for these two types of experiments. Once again, your instructor may have a particular style that is recommended, but we provide suggested formats below.

Notebook Format for Investigative Experiments

1. **Heading.** Use a new page of the notebook to start the entries for the experiment. Provide information that includes your name, the date, the title of the experiment, and a reference to the place in the laboratory textbook or other source where the procedure may be found.
2. **Introduction.** Give a brief introduction to the experiment in which you clearly state the purpose(s) of the procedure. This should require no more than one-fourth of a page.
3. **Summary of MSDS Data.** As directed by your instructor, either briefly summarize the Material Safety Data Sheet (MSDS) data (Sec. 1.10) for the solvents, reagents, and products encountered in the experiment or give a reference to where a printout of these data is located.
4. **Synopsis of and Notes on Experimental Procedure—Results.** Enter a one- or two-line statement for each part of an experiment. Reserve sufficient room to record results as they are obtained. As noted in Section 1.5 of "Notebook Format for Preparative Experiments," do *not* copy the experimental procedure from the textbook, but provide a synopsis of it.

   Much of this section of the write-up can be completed before coming to the laboratory, to ensure that you understand the experiment and that you will perform all parts of it.
5. **Interpretation of Instrumental Data.** If instructed to do so, discuss any instrumental data, such as gas-liquid chromatographic analyses and spectral data that you have obtained or are provided in the textbook.
6. **Conclusions.** Record the conclusions that can be reached, based on the results you have obtained in the experiment. If the procedure has involved identifying an unknown compound, summarize your findings in this section.
7. **Answers to Exercises.** Enter answers to any exercises for the experiment that have been assigned from the textbook.

A sample write-up of an investigative experiment is given in Figure 1.1.

Notebook Format for Preparative Experiments

1. **Heading.** Use a new page of the notebook to start the entries for the experiment. Provide information that includes your name, the date, the title of the experiment, and a reference to the place in the laboratory textbook or other source where the procedure may be found.
2. **Introduction.** Give a brief introduction to the experiment in which you clearly state the purpose(s) of the procedure. This should require no more than one-fourth of a page.
3. **Main Reaction(s) and Mechanism(s).** Write *balanced* equations giving the main reaction(s) for conversion of starting material(s) to product(s). The reason for balancing the equations is discussed in Part **4 Table of Reactants and Products**. Whenever possible, include the detailed mechanisms for the reactions that you have written.
4. **Table of Reactants and Products.** Set up a Table of Reactants and Products as an aid in summarizing the amounts and properties of reagents and catalysts being used and the product(s) being formed. Only those reactants, catalysts, and

1. **Your Name**
Date

Separation of Green Leaf Pigments by TLC

Reference: *Experimental Organic Chemistry: A Miniscale and Microscale Approach,* 6th ed., by Gilbert and Martin, Section 6.2.

2. INTRODUCTION

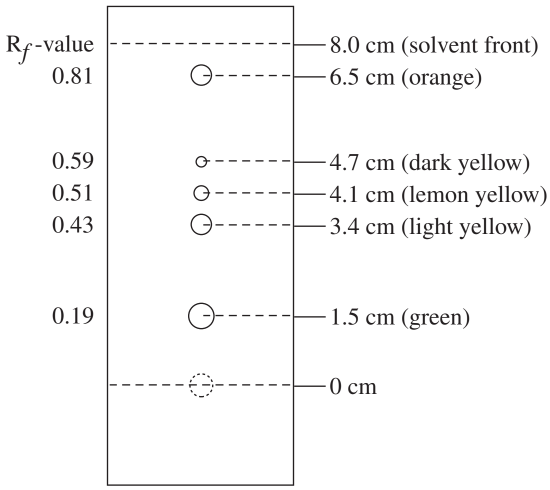
The pigments in green leaves are to be extracted into an organic solvent, and the extract is to be analyzed by thin-layer chromatography (TLC). The presence of multiple spots on the developed TLC plate will indicate that more than a single pigment is contained in the leaves.

3. MSDS DATA

These data are available on the printouts inserted at the back of my lab book.

4. SYNOPSIS OF AND NOTES ON EXPERIMENTAL PROCEDURE—RESULTS

Procedure: Grind five stemless spinach leaves in mortar and pestle with 5 mL of 2:1 pet. ether and EtOH. Swirl soln. with 3 × 2-mL portions H_2O in sep. funnel; dry org. soln. for few min over anhyd. Na_2SO_4 in Erlenmeyer. Decant and concentrate soln. if not dark-colored. Spot 10-cm × 2-cm TLC plate about 1.5 mm from end with dried extract; spot should be less than 2 mm diam. Develop plate with $CHCl_3$. *Variances and observations:* Procedure followed exactly as described in reference. Org. soln. was dark green in color; aq. extracts were yellowish. Half of org. layer lost. TLC plate had five spots having colors and R_f-values shown on the drawing below.



5. INTERPRETATION OF INSTRUMENTAL DATA

No data provided for this experiment.

6. CONCLUSIONS

Based on TLC analysis, the procedure used allows the extraction of at least five different pigments from the spinach leaves. Judging from colors, one of these is a carotene, three are xanthophylls, and the last is chlorophyll *b*.

7. ANSWERS TO EXERCISES

(Answers intentionally omitted.)

Figure 1.1
Sample notebook format for investigative experiments.

products that appear in the main reaction(s) should be listed in the table; many other reagents may be used in the work-up and purification of the reaction mixture, but these should *not* be entered in the table.

Your instructor will have specific recommendations about what should appear in the table, but the following items are illustrative.

a. The name and/or structure of each reactant, catalyst, and product.

b. The molar mass of each compound.

c. The weight used, in grams, of each reactant and the volume of any liquid reactant. We recommend that the weight and/or volume of any catalysts used be entered for purposes of completeness.

d. The molar amount of each reactant used; this can be calculated from the data in Parts **b** and **c.**

e. The theoretical mole ratio, expressed in whole numbers, for the reactants and products; this ratio is determined by the *balanced* equation for the reaction, as given in Part **3**, Main Reaction(s) and Mechanism(s).

f. Physical properties of the reactants and products. This entry might include data such as boiling and/or melting point, density, solubility, color, and odor.

g. As directed by your instructor, either briefly summarize the MSDS data (Sec. 1.10) for the solvents, reagents, and products encountered in the experiment or give a reference to where a printout of these data is located.

5. **Yield Data.** Compute the maximum possible amount of product that can be formed; this is the **theoretical yield.** This can easily be calculated from the data in the Table of Reactants and Products as follows. First determine which of the reactants corresponds to the **limiting reagent.** This is the reagent that is used in the *least* molar amount relative to what is required theoretically. In other words, the reaction will stop once this reactant is consumed, so its molar quantity will define the maximum quantity of product that can be produced. From the number of moles of limiting reagent involved and the balanced equation for the reaction, determine the theoretical yield, in moles (written as "mol" when used as a unit, as in "g/mol"), of product. This value can then be converted into the theoretical yield in grams, based on the molar mass of the product.

   Once the isolation of the desired product(s) has been completed, you should also calculate the **percent yield,** which is a convenient way to express the overall efficiency of the reaction. This is done by obtaining the **actual yield** of product(s) in grams, and then applying the expression in Equation 1.1. Generally, the calculated value of percent yield is rounded to the nearest whole number. As points of reference, most organic chemists consider yields of 90% or greater as being "excellent," and those below 20% as "poor."

$$\text{Percent yield} = \frac{\text{Actual yield (g)}}{\text{Theoretical yield (g)}} \times 100 \tag{1.1}$$

6. **Synopsis of and Notes on Experimental Procedure.** Provide an outline of the experimental procedure that contains enough detail so that you do not have to refer to the textbook repeatedly while performing the experiment. Note any variations that you use, as compared to the referenced procedure, and observations that you make while carrying out the formation and isolation of the product(s).

7. **Observed Properties of Product.** Record the physical properties of the product that you have isolated in the experiment. Appropriate data under this heading might include boiling and/or melting point, odor, color, and crystalline form, if the product is a solid. Compare your observations with those available on the compound in various reference books (e.g., the *CRC Handbook of Chemistry and Physics* or *Lange's Handbook of Chemistry*).

8. **Side Reactions.** If instructed to do so, list possible side reactions (those reactions leading to undesired products) that are likely to occur in the experiment. It is important to consider such processes because the by-products that are formed must be removed by the procedure used to purify the desired product. You may need to consult your lecture notes and textbook to predict what side reactions might be occurring.

9. **Other Methods of Preparation.** If instructed to do so, suggest alternative methods for preparing the desired compound. Such methods may involve using entirely different reagents and reaction conditions. Your lecture notes and textbook can serve as valuable resources for providing possible entries for this section.

10. **Method of Purification.** Develop a flowchart that summarizes the sequence of operations that will be used to purify the desired product. The chart will show at what stages of the work-up procedure unchanged starting materials and unwanted by-products are removed. By understanding the logic of the purification process, you will know why each of the various operations specified in the purification process is performed.

    Purifying the final product of a reaction can be the most challenging part of an experimental procedure. Professional organic chemists are constantly required to develop work-up sequences that allow isolation of a pure product, free from starting materials and other contaminants. They do this by considering the chemical and physical properties of both the desired and undesired substances, and it is important for you to gain experience in devising such schemes as well.

11. **Interpretation of Instrumental Data.** If instructed to do so, discuss any instrumental data, such as gas-liquid chromatographic analyses and spectral data you have obtained or that are provided in the textbook.

12. **Answers to Exercises.** Enter answers to any exercises for the experiment that have been assigned from the textbook.

A detailed example of the write-up for a preparative experiment involving the dehydration of cyclohexanol (Sec. 10.3) is given in Figure 1.2. You may not actually perform this reaction; nevertheless, you should carefully study the example to see how to prepare specific entries for the first eight items listed. The various entries in Figure 1.2 are labeled with circled, **boldface** numbers and are discussed further in the following paragraphs. It is assumed for illustrative purposes that an actual yield of 2.7 g is obtained.

1 Use a new page of the notebook to start the entries for the experiment. Provide information that includes your name, the date, the title of the experiment, and a reference to the place in the laboratory textbook or other source where the procedure can be found.

2 Self-explanatory.