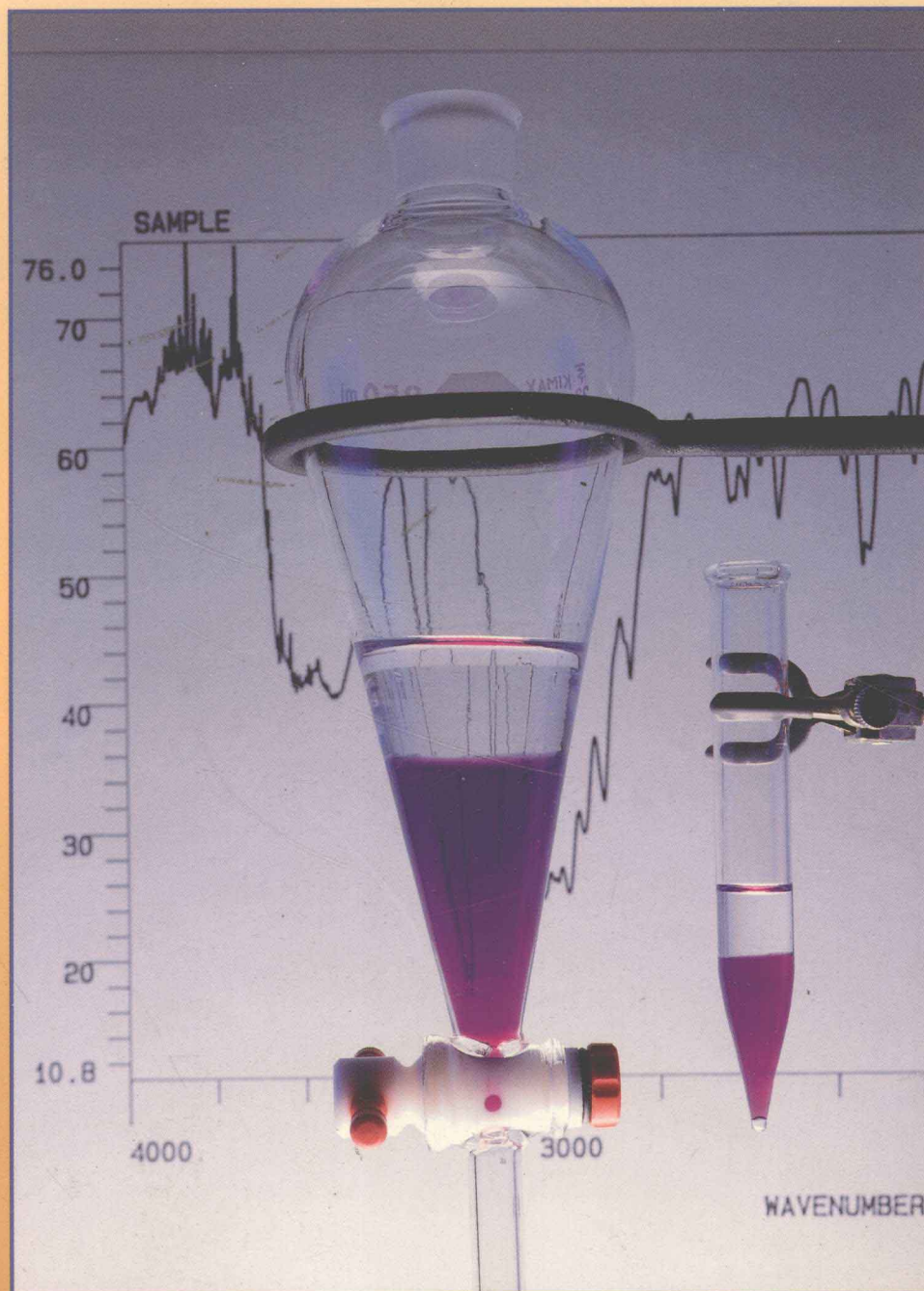


# ORGANIC CHEMISTRY LABORATORY

Standard & Microscale Experiments



RODIG / BELL / CLARK

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# Organic Chemistry Laboratory

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## Standard and Microscale Experiments



**SAUNDERS COLLEGE PUBLISHING**

Philadelphia Ft. Worth Chicago San Francisco  
Montreal Toronto London Sydney Tokyo

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Text Typeface: Times Roman  
Compositor: EPS Group, Inc.  
Acquisitions Editor: John Vondeling  
Developmental Editor: Kate Pachuta  
Managing Editor: Carol Field  
Project Editor: Janet B. Nuciforo  
Copy Editor: Charlotte Nelson  
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Cover Designer: Lawrence R. Didona  
Text Artwork: Rolin Graphics Inc.  
Director of EDP: Tim Frelick  
Production Manager: Bob Butler

Cover Credit: Photograph by Robert K. Ander

Printed in the United States of America

ORGANIC CHEMISTRY LABORATORY      Standard and Microscale Experiments

0-03-012644-4

Library of Congress Catalog Card Number: 89-043220

0123 071 987654321

# Preface

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In recent years, there has been a growing tendency to carry out elementary organic laboratory experiments on a so-called “micro” scale; that is, to use milligram quantities of materials rather than grams. The advantages of such small scale experiments are obvious: the use of smaller amounts of materials, normally a major cost item in such courses; generally shorter reaction times; greatly diminished waste disposal problems; lower potential safety hazards; and a practical accession to experiments utilizing relatively expensive starting materials.

Recently, several laboratory texts have appeared which are structured around the microscale concept. We have felt strongly for some time that a well-rounded training course in elementary organic laboratory principles should include certain “macro” sized experiments since techniques used in these are often quite different from those using micro quantities. Thus, students should certainly learn techniques such as distillation, extraction, and filtration, and a sampling of synthetic procedures on a larger scale since they surely will meet such situations in the typical organic laboratory they may encounter in their future.

In this book, we describe three types of experiments—those that teach valuable techniques but are not practical on a very small scale, those that can be carried out on either scale and where the choice is left to the instructor or the student; and finally those that are practical only on a micro scale, generally because of the prohibitive costs of materials or excessively long reaction times if carried out in larger quantities. By far, the largest number of experiments are of the dual type.

In general, the microscale experiments were developed using what we consider practical amounts of materials. At times, the scale may be slightly larger than those described by others, but our experiences have shown that with very small amounts, the slight additional saving in chemical costs did not justify the increased frustrations the students experienced when working

with such small quantities. In addition, the microscale procedures are based on techniques used for many years in research laboratories, and can be carried out using readily available standard laboratory equipment, such as the centrifuge and test tube, Pasteur pipet, Hirsch funnel, septum, syringe, and ordinary small-scale glassware, thus eliminating the need for specialized apparatus or micro kits.

We have also attempted to include some more unusual experiments, such as Aromatic Nucleophilic Substitution (Chapter 26), Heterocyclic Syntheses (Chapter 37), a comparison of Acid Catalyzed and Enzymatic Hydrolyses (Chapter 42), and the Analysis of Fats and Oils (Chapter 43). A discussion of  $^{13}\text{C}$  NMR is included in Chapter 10, and on-line literature searching in Chapter 11. To increase a student's exposure to instrumentation, we describe experiments utilizing gas chromatography in Chapters 6, 13, 16, and 43, and the optional use of high performance liquid chromatography (HPLC) in Chapter 13. The use of a pH meter is included in Chapter 22, a visible spectrometer or colorimeter in Chapter 34, and a simple polarimeter in Chapters 39 and 42. In the macroscale experiments, we have attempted to use products from one experiment as the starting material for another where practical, thus further reducing chemical costs and waste disposal problems.

Safety is all-important in any laboratory course. It is stressed in a general section in Chapter 1 and throughout the text in the form of **Safety Notes** associated with each experiment. Whenever possible, chemicals classed as hazardous by OSHA have been eliminated. In the few instances where they do appear, they and the associated hazards are clearly identified so that adequate safety precautions can be taken. Further advice on the safe dispensing of reagents and other general information on the experiments can be found in the accompanying Instructors Manual.

A difficulty frequently encountered in organic laboratory texts is that early experiments do not correlate well with material covered in the lecture course. To avoid this problem, the first eight chapters emphasize laboratory techniques, followed by two on instrumentation and one on literature searching. Only then are experiments described where an understanding of the chemistry makes the laboratory experience most meaningful.

A number of the experiments contain unknowns; thus the answers are not forthcoming until the experiments are performed. We find this approach heightens student interest and emphasizes the importance of working carefully to obtain the correct result.

In any laboratory course, the student's work efficiency is greatly enhanced if he or she ponders the material beforehand. To this end, we have included prelaboratory questions for all chapters except 1 and 46. In addition, we have added questions of variable difficulty at the ends of the chapters, which can be best answered after the experiments have been completed.

All of the experiments can be carried out in one (or two) three-hour laboratory period, and have been repeatedly tested in our classes at the University of Virginia and Old Dominion University. In this regard, we wish to express our gratitude to Robin E. Fretwell, Andrea L. Cox, Rashmi A. Majali, Charles G. diPierro, Broderick C. Bello, and Joseph C. Pedulla, who spent considerable time and effort in developing the microscale procedures. Finally, we would like to thank Professors Exum D. Watts, of Middle Tennessee State University; Charles E. Sundin, of the University of Wisconsin, Platteville; Kenneth W. Raymond, of Eastern Washington University; Lisa McElwee-White, of Stanford University; and Arlyn Myers, of the University of California, Berkeley for their helpful comments and criticisms; Project Editor Janet Nuciforo for her patience and helpful suggestions during the preparation of the manuscript; and John Vondeling, Kate Pachuta, and others on the staff of Saunders College Publishing, for their help, cooperation, and encouragement.

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**Charles E. Bell, Jr.**  
**Allen K. Clark**

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# 1

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## Introduction

Laboratory work is an integral and essential part of any chemistry course. Chemistry is an experimental science—the compounds and reactions that are met in lecture and classroom work have been discovered by *experimental observations*. Organic compounds exist as gases, liquids, or solids with characteristic odors and physical properties. They are synthesized, distilled, crystallized and chromatographed, and then transformed by reactions into other compounds. The purpose of laboratory work is to provide an opportunity to observe the reality of compounds and reactions and to learn something of the operations and techniques that are used in experimental organic chemistry and in other areas in which organic compounds are encountered.

### **LABORATORY SAFETY**

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Along with the opportunity to learn at first hand about the properties and reactions of compounds and the manipulation of laboratory equipment, there must be proper concern for safety. Most organic compounds are flammable, and they are toxic or irritating to a greater or lesser degree. Many organic reactions are potentially violent. Laboratory work in organic chemistry is not a dangerous occupation, however, nor is the laboratory a perilous place to be, provided that some simple precautions and safety rules are followed. There are some potential hazards that must be recognized and avoided; accidents can and do occur when these hazards are ignored. Throughout this book, **Safety Notes** are included for each experiment. They emphasize the specific hazards that may be encountered and must be kept in mind. In the following section, some general precautions are discussed, and a few rules are given that must be observed during any laboratory work.

### **RULES FOR PERSONAL SAFETY**

Avoiding injuries is largely a matter of good sense. Carelessness can lead to accidents and injuries to yourself and others. The following rules cover some important general precautions and should be observed at all times.

1. **Eye Protection.** Approved eye protection must be worn at all times in the laboratory, regardless of what is being done. In many locations, chemical safety goggles are required by law. Safety glasses (either prescription or plain) with side shields to protect from splashes also offer good eye protection.

Contact lenses should *not* be worn in the laboratory. If a splash occurs, the lens can act as a trap for corrosive materials and greatly increase the likelihood of permanent injury. Moreover, low levels of vapors that are present in the laboratory can cause severe irritation by the lens.

2. Never work in a laboratory without another person being present or within calling distance. Minor accidents can become disasters if help is not available.
3. Do not carry out any reaction that is not specifically authorized by the instructor.
4. Never taste a compound; never pipet a chemical by mouth; do not eat, drink, or smoke in the laboratory.
5. Avoid contact of the skin with any chemical. If a substance is spilled on your hands, wash them thoroughly with soap and water. Do not rinse them with a solvent, since this may cause more rapid absorption.
6. Long hair should be tied back. Shoes must be worn to prevent injury from spilled chemicals or bits of glass. Avoid loose-fitting sleeves and clothing that leave expanses of skin unprotected.
7. Never heat a flask or any apparatus that is sealed or stoppered (i.e., a closed system)—make certain that there is an opening to the atmosphere.
8. When inserting glass tubing into a rubber stopper or rubber tubing, lubricate it first with a drop of glycerin and protect your hands with a towel. The same is true for inserting thermometers into stoppers. Thin-walled transfer pipets should not be used as connectors for rubber tubing or stoppers; they are fragile and very easily crushed.
9. Some experiments require the use of a well-ventilated hood. These experiments should not be attempted in the open laboratory.
10. **Peroxides.** A major safety concern with certain organic substances is the buildup of potentially explosive peroxides. These can form by the light-catalyzed autoxidation of ethers, aldehydes, and alkenes and aromatic compounds having allylic or benzylic hydrogen atoms, and even alcohols. The danger is particularly acute when these materials are used as reaction solvents or in extractions and then are concentrated in the workup. This is one reason why liquids should never be distilled to dryness. Such commonly used substances as the diethyl and diisopropyl ethers, tetrahydrofuran, cumene, tetralin, and 2-butanol (Fig. 1.1) should be checked for peroxides if they have been stored for an extended period of time, especially in a partially empty container. Particular care

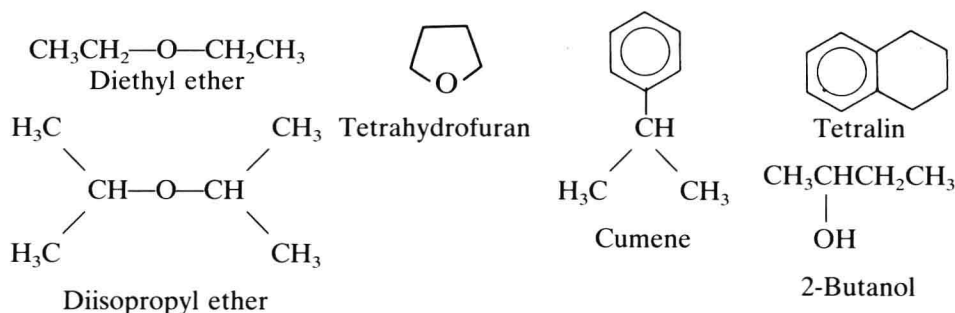


Figure 1.1 Some substances that can form peroxides by autoxidation.

should be taken with diethyl ether because of its wide use as an extraction solvent.

Peroxides can be detected with starch iodide paper or by adding 1 mL of the suspected material to 1 mL of glacial acetic acid containing 0.1 g of sodium or potassium iodide. A yellow-to-brown color indicates the presence of peroxides. A blank should be run to confirm the validity of the test.

Peroxides can be removed from ethers by shaking with a 30% solution of aqueous ferrous sulfate or by percolating through a column of alumina. The latter method also removes traces of water. Additional information on peroxides can be found in the references cited at the end of the chapter.

## FIRST AID

- 1. Emergency Equipment.** Learn the location of safety showers, eyewash fountains, and fire extinguishers, and know how this equipment is used.
- 2. Chemical Spills on the Skin.** Immediately flush the skin with running water for several minutes; if the eyes or face are involved, use an eyewash fountain or the nearest faucet. For minor acid burns, use a paste or ointment of sodium bicarbonate after the flushing. For bromine, apply a compress soaked in 0.6 M sodium thiosulfate solution. For any severe chemical burn, consult a physician as soon as possible after the initial thorough water flushing.
- 3. Fire.** If clothing is ignited, immediately extinguish it with a safety shower, by rolling on the floor, or, if necessary, with a coat or anything else available. Do not allow a person with burning clothing to run—such action only fans the flames. Even standing should be avoided because of the danger of inhaling hot fumes.\*

\*Fire blankets are no longer recommended for use with fires. They can act as a chimney, thus encouraging the fire. Such blankets can be useful, however, for covering victims in shock.

4. In any severe case of chemical spill, a burn, or a cut, the affected person should be escorted to a physician or a hospital emergency room.

## CHEMICAL TOXICITY AND CARCINOGENS

It has long been recognized that certain chemicals—for example, phosgene or the nerve gas isopropyl methylfluorophosphonate—are highly toxic substances that are lethal in extremely small amounts. In recent years there has been a considerable increase in awareness and concern about the toxicity of all chemicals encountered in laboratory and manufacturing environments. Major efforts are now being made to identify toxic chemicals and avoid exposure to them.

The National Institute of Occupational Safety and Health (NIOSH) has prepared a registry of a large number of compounds for which some data on toxic effects are available. Another government agency, the Occupational Safety and Health Administration (OSHA), issues regulations governing permissible limits of exposure to chemicals, with particular attention being placed on compounds that are commonly encountered as air contaminants. Many of the compounds for which limits have been set, such as diethyl ether and ethanol, have relatively low toxicity, but limits are nevertheless placed on prolonged exposure. On the other hand, compounds that may have high acute toxicity can be transferred and used in a laboratory experiment with simple precautions to avoid contact.

A major concern in recent years has been the carcinogenicity of organic compounds, that is, their ability to induce cancer. For certain compounds that were used industrially for many years, there is a clear link between exposure to the compound and the incidence of certain types of cancer in the workers who handled them, just as there is between cigarette smoking and lung cancer. More recently, evidence has been found for the occurrence of tumors in experimental animals exposed to very large doses of a wide variety of other organic compounds. A much-publicized example is the artificial sweetener saccharin, which was used for many years in low-calorie beverages.

Government agencies, such as the Carcinogen Assessment Group (CAG) of the Environmental Protection Agency (EPA) and NIOSH have compiled lists of suspected carcinogens, and laboratory chemical catalogs often identify these as “cancer suspect agents.” The National Institutes of Health has published guidelines for the laboratory use of certain chemical carcinogens, and OSHA, in turn, has issued regulations on exposure to a number of carcinogenic substances, most of which are in commercial use.

A brief list of carcinogenic compounds that are frequently encountered in laboratory work is given in Table 1.1. It should be pointed out that this list contains only a very small fraction of the compounds for which data on carcinogenic properties are known. Remember that carcinogenic activity is usually based on tests in animals at high doses for prolonged duration, and

**Table 1.1 Partial List of Chemical Carcinogens**

acetamide	$\text{CH}_3\text{CONH}_2$	dimethyl sulfate	$(\text{CH}_3)_2\text{SO}_4$
acrylonitrile	$\text{CH}_2=\text{CHCN}$	dioxane	$\text{O}(\text{CH}_2\text{CH}_2)_2\text{O}$
aminobiphenyl	$\text{NH}_2\text{C}_6\text{H}_4\text{C}_6\text{H}_5$	ethyl carbamate	$\text{NH}_2\text{CO}_2\text{C}_2\text{H}_5$
aziridine	$\text{CH}_2\text{CH}_2\text{NH}$	hydrazine	$\text{NH}_2\text{NH}_2$
benzene	$\text{C}_6\text{H}_6$	methyl iodide	$\text{CH}_3\text{I}$
benzidine	$\text{NH}_2\text{C}_6\text{H}_4\text{C}_6\text{H}_4\text{NH}_2$	1-naphthylamine	$1\text{-C}_{10}\text{H}_7\text{NH}_2$
benzpyrene	$\text{C}_{20}\text{H}_{12}$	2-naphthylamine	$2\text{-C}_{10}\text{H}_7\text{NH}_2$
bis(chloromethyl) ether	$(\text{ClCH}_2)_2\text{O}$	4-nitrobiphenyl	$\text{NO}_2\text{C}_6\text{H}_4\text{C}_6\text{H}_5$
<i>t</i> -butyl chloride	$(\text{CH}_3)_3\text{CCl}$	nitrosomethylurea	$\text{NH}_2\text{CON}(\text{CH}_3)\text{NO}$
carbon tetrachloride	$\text{CCl}_4$	phenylhydrazine	$\text{C}_6\text{H}_5\text{NHNH}_2$
chloroform	$\text{CHCl}_3$	thiourea	$\text{NH}_2\text{CSNH}_2$
chromic anhydride	$\text{CrO}_3$	<i>o</i> -toluidine	$\text{CH}_3\text{C}_6\text{H}_4\text{NH}_2$
diazomethane	$\text{CH}_2\text{N}_2$	trichloroethylene	$\text{CHCl}=\text{CCl}_2$
dibromoethane	$\text{BrCH}_2\text{CH}_2\text{Br}$	vinyl chloride	$\text{CH}_2=\text{CHCl}$

the risk from occasional brief exposure is unknown. The chief concern with carcinogens, as with other toxic substances, is with permissible levels for continuous exposure, not occasional use. The important consideration for laboratory work is to avoid any unnecessary exposure and to use these compounds only when essential, with due care and protection. In particular, benzene, chloroform, and carbon tetrachloride should not be used as solvents for extraction or column chromatography.

In the experiments in this book, all compounds that have been implicated as carcinogens have been eliminated wherever possible; in the remaining few cases they have been identified as such. It must be emphasized that, with present knowledge, these compounds should *not* be considered as deserving any more concern than many others. Careless handling of simple acids or solvents normally presents a much greater safety hazard.

## DISPOSAL OF CHEMICALS

In recent years there has been an ever-increasing awareness of the pollution of our water supplies. Many areas have strict laws and/or regulations concerning the disposal of chemicals in sinks. It is best to check with local authorities before disposing of *any* chemicals in sinks. Water-immiscible solvents or other organic liquids should be discarded in a designated waste solvent container and should not be poured into a sink. Residues and vapors can remain in the sink trap; moreover, any chemicals discharged in a drain eventually add to pollution of surface or ground water.

Chemicals that react vigorously with water, such as acid chlorides, metal hydrides, or alkali metals, should be decomposed in a hood in a

suitable way, such as by controlled reaction with alcohol. Strong acids or strong bases should be neutralized before they are discarded.

Solids or glass should be disposed of in a nonmetallic chemical waste jar; do not throw them into a wastepaper basket.

## **FIRE HAZARDS**

Fire hazards are present in any organic laboratory because of the frequent use of volatile, flammable solvents. By far the greatest risk of fire is associated with gas burners for heating, and this form of heating should be avoided whenever possible. On the other hand, electric heating devices may contain exposed elements and are not totally immune from being fire hazards.

Organic vapors are heavier than air and flow downward; they diffuse rapidly and can be ignited several feet away from the source. The following precautions should always be observed:

1. Organic liquids should only be heated to boiling or distilled on a steam bath or under a condenser. When refluxing a liquid, particularly over a flame, be certain that the condenser is tightly fitted to the flask.
2. Before lighting a flame, if one must be used, check to see that volatile liquids are not being poured or evaporated in your vicinity. Conversely, before pouring or evaporating a liquid, be certain that none of your neighbors is using a flame. Always turn off a burner as soon as you are finished using it—never leave it on unnecessarily.
3. Smoking creates an avoidable fire hazard and is not permitted in the laboratory.

## **LABORATORY EQUIPMENT AND TECHNIQUES**

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For successful laboratory work it is essential that before beginning an experiment you understand what you are going to do and why and how you are going to do it. Study the assigned experiment in advance and plan your operations. If required by your instructor, answer in writing the prelab questions at the end of the experiment. These questions provide background for the work and will acquaint you with points that can and should be understood before you actually do the experiment.

Experimentation in organic chemistry calls into play a number of operations and techniques and a rather large assortment of apparatus. Detailed instructions in the techniques and equipment used for various separation and purification methods are given in Chapters 2 through 8 in conjunction with actual experimental procedures. A few points of general practice that apply to nearly all experiments are covered in this section.

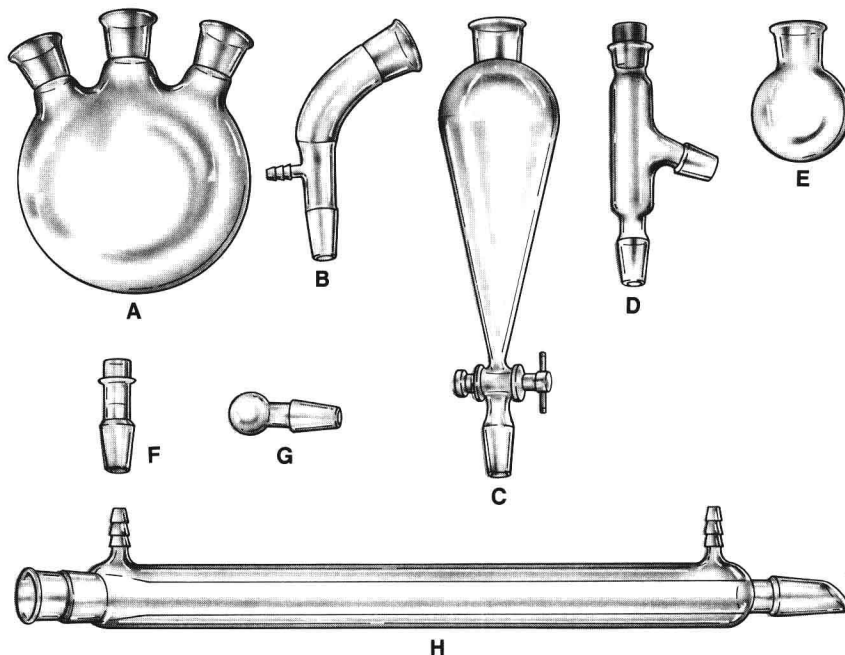
## EQUIPMENT

### Glassware

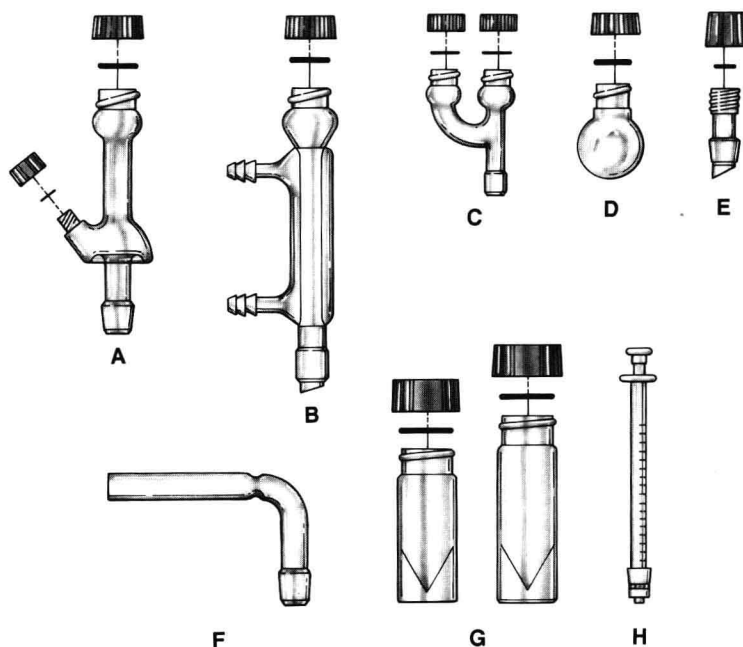
In this book you will learn two major laboratory techniques for carrying out chemical reactions. Those on a *macroscale* normally involve gram quantities of materials, and typical glassware used for this is shown in Figure 1.2. The glassware is equipped with standard-taper ground joints that permit quick and secure assembly of apparatus. It is expensive and must be handled with care. *Microscale* experiments are carried out on milligram amounts of material and generally demand more careful manipulation of reagents and products. Often these experiments can also be carried out with the glassware shown in Figure 1.2, but on a reduced scale.

Some examples of glassware designed specifically for microscale use are shown in Figure 1.3. This glassware contains joints with external threads and open-top caps that allow them to be screwed together. Since the pieces cannot pull apart, the assemblies require considerably less clamping. In addition, the inner parts of the joints are also standard-taper ground glass that allow their attachment to other ground joint apparatus. Although convenient, such equipment is not required for the experiments in this book.

Other equipment that will be needed include Erlenmeyer flasks, beakers, and test tubes in various sizes, Buchner & Hirsch funnels, graduated



**Figure 1.2** Standard-taper glassware. (A) three-necked round-bottom flask; (B) vacuum take-off adapter; (C) dropping funnel; (D) distillation head with rubber connector; (E) round-bottom flask; (F) tubing connector; (G) standard-taper stopper; (H) condenser.



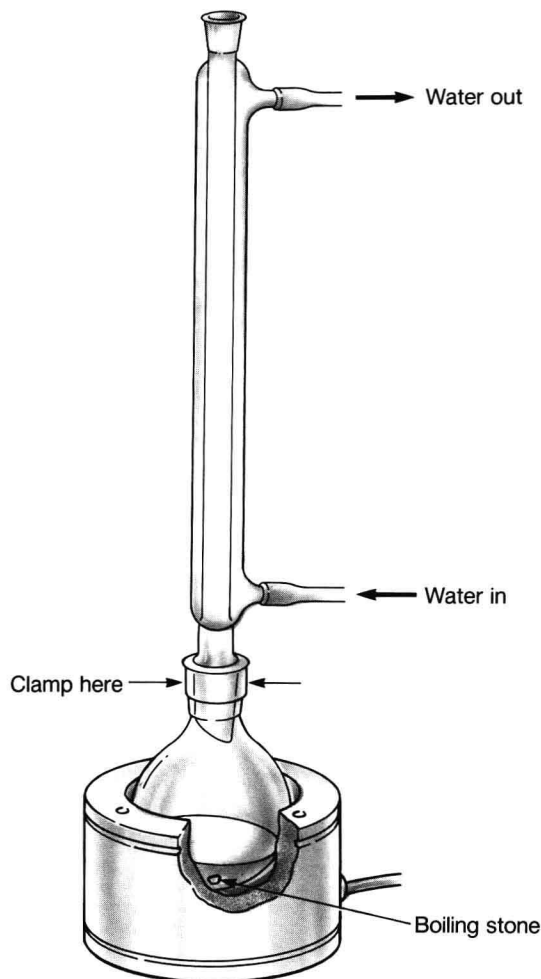
**Figure 1.3** Microscale glassware. (A) Hickman still head; (B) condenser; (C) Claisen head adapter; (D) small round-bottom flask; (E) thermometer adapter; (F) drying tube; (G) 3- and 5-mL conical reaction vials; (H) 1-mL sample syringe.

cylinders, pipets, centrifuge tubes (preferably graduated), thermometers, spatulas, septums, syringes, and assorted clamps and other hardware.

For effective laboratory work it is most important that you develop good working habits, learn the proper equipment for a given purpose, and know how to use it. Maintain a well-organized locker or equipment drawer; keep your equipment clean and as conveniently located as possible. Make a practice of washing or rinsing glassware as soon as it has been used so that it will be clean and dry the next time you need it. Take enough time to clean up and store equipment properly before you leave the laboratory.

### Reaction Setups

Many reactions involve the combining of two reactants at a controlled rate. Simple reactions on a small scale require only a pipet and centrifuge or test tube, which can be swirled by hand. In other cases, on both macroscale and microscale, the reactants are combined and heated in a simple setup, such as that shown in Figure 1.4. The temperature is controlled by a heating unit or by the boiling point of a reactant or solvent. Mixing, if needed, is provided by the turbulence of boiling. If the reaction is exothermic, cooling occurs by the heat being transferred to the condenser by the refluxing solvent, or, if necessary, the flask can be immersed in an ice bath.



**Figure 1.4** Simple reflux apparatus.

For reactions on a large scale or those requiring special conditions, a more elaborate setup such as that shown in Figure 1.5 may be needed. The separate necks for the dropping funnel, stirrer, and condenser permit flexible assembly and easy control of addition and mixing.

Stirring is important if a reactant must be added and dispersed at a steady rate or if the reaction must occur between separate phases. For relatively small flasks and for reaction mixtures that are not viscous, a bar magnet with an inert coating is placed in the flask and is spun by a motor-driven magnet below the flask (Fig. 1.6). Alternatively (as seen in Fig. 1.5), a rod with a small propeller or paddle, turning in a closely fitting sleeve, is driven by an electric or air-powered motor.

The atmosphere in a reaction may be critical. If the reactants or products are sensitive to water, a drying tube filled with desiccant attached to the condenser may be sufficient. For scrupulous removal of moisture or