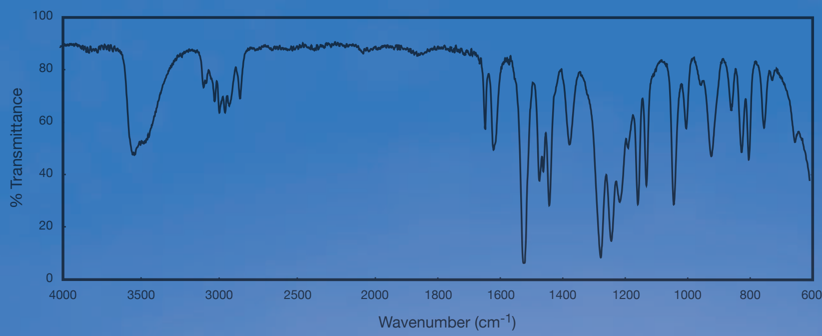
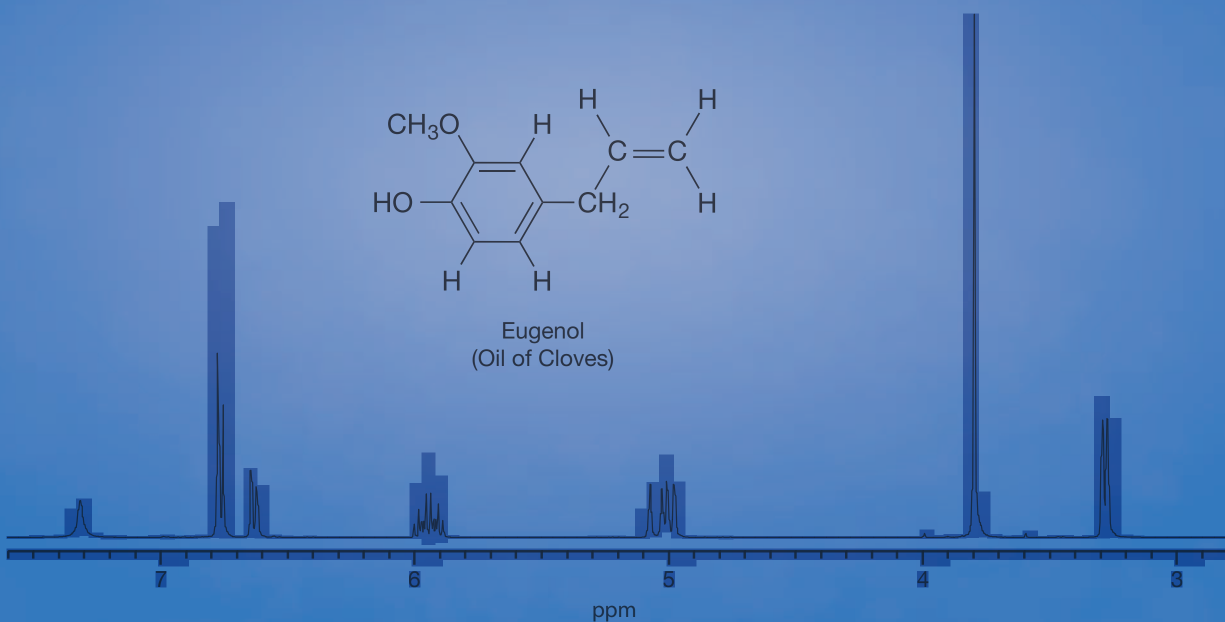


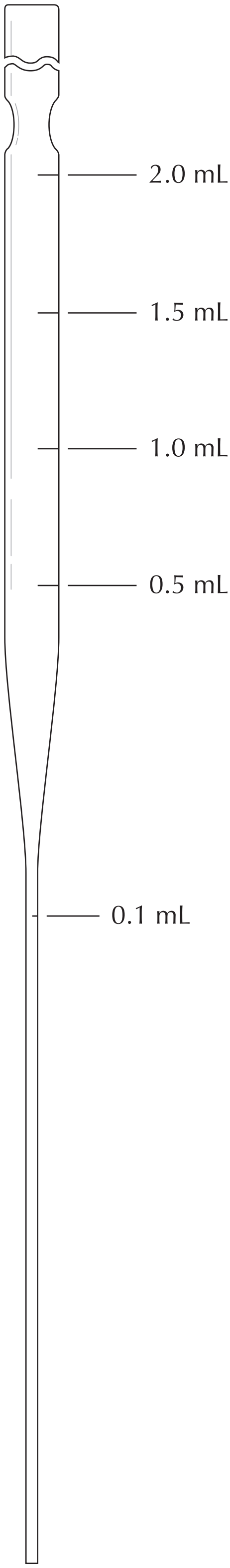


Laboratory Techniques in Organic Chemistry

FOURTH EDITION

Supporting Inquiry-Driven Experiments

Jerry R. Mohrig
David G. Alberg
Gretchen E. Hofmeister
Paul F. Schatz
Christina Noring Hammond



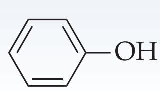
Chemical resistance of common types of gloves to various compounds

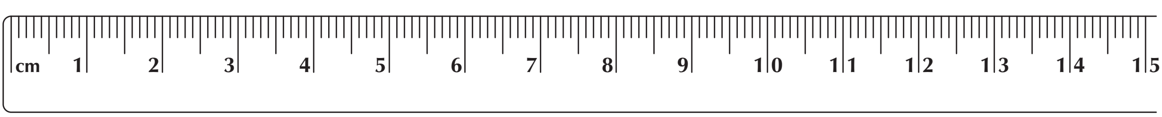
Compound	Glove type: Neoprene	Nitrile	Latex
Acetone	Good	Fair	Good
Chloroform	Good	Poor	Poor
Dichloromethane	Fair	Poor	Poor
Diethyl ether	Very good	Good	Poor
Ethanol	Very good	Good	Good
Ethyl acetate	Good	Poor	Fair
Hexane	Excellent	Excellent	Poor
Hydrogen peroxide	Excellent	Good	Good
Methanol	Very good	Fair	Fair
Nitric acid (conc.)	Good	Poor	Poor
Sodium hydroxide	Very good	Good	Excellent
Sulfuric acid (conc.)	Good	Poor	Poor
Toluene	Fair	Fair	Poor

Common organic solvents

Name	Boiling point (°C)	Density (g/mL)	Dielectric constant	Miscible with H_2O
Acetone (2-propanone)	56.5	0.792	21	yes
Dichloromethane	40	1.326	9.1	no
Diethyl ether	35	0.713	4.3	no
Ethanol (95% aq. azeotrope)	78	0.816	27	yes
Ethanol (anhydrous)	78.5	0.789	25	yes
Ethyl acetate	77	0.902	6.0	slightly
Hexane	69	0.660	1.9	no
Methanol	65	0.792	33	yes
Pentane	36	0.626	1.8	no
2-Propanol (isopropyl alcohol)	82.5	0.785	18	yes
Toluene	111	0.866	2.4	no

Selected approximate pK_a values

Name	Formula	pK_a	Name	Formula	pK_a
Sulfuric acid	H_2SO_4	−5	Ammonium	NH_4^+	10
Aqueous mineral acids	H_3O^+	−2	Protonated amines	R_3NH^+	10
Carboxylic acids	$R\overset{\overset{O}{\|}}{C}OH$	5	Alcohols	ROH	16
Bicarbonate	HCO_3^-	6	Water	H_2O	16
Phenols		10	Acetone	$CH_3\overset{\overset{O}{\|}}{C}CH_3$	19



PERIODIC TABLE OF THE ELEMENTS

	1 I IA	2 II IIA	3 IIIB	4 IVB	5 VB	6 VIB	7 VIIB	8 VIIIB	9 VIIIB	10 VIIIB	11 IB	12 IIB	13 III IIIA	14 IV IVA	15 V VA	16 VI VIA	17 VII VIIA	18 VIII VIIIA
1	1 **H** 1.0079																	2 **He** 4.00
2	3 **Li** 6.94	4 **Be** 9.01											5 **B** 10.81	6 **C** 12.01	7 **N** 14.01	8 **O** 16.00	9 **F** 19.00	10 **Ne** 20.18
3	11 **Na** 22.99	12 **Mg** 24.31											13 **Al** 26.98	14 **Si** 28.09	15 **P** 30.97	16 **S** 32.06	17 **Cl** 35.45	18 **Ar** 39.95
4	19 **K** 39.10	20 **Ca** 40.08	21 **Sc** 44.96	22 **Ti** 47.88	23 **V** 50.94	24 **Cr** 52.00	25 **Mn** 54.94	26 **Fe** 55.85	27 **Co** 58.93	28 **Ni** 58.71	29 **Cu** 63.54	30 **Zn** 65.37	31 **Ga** 69.72	32 **Ge** 72.59	33 **As** 74.92	34 **Se** 78.96	35 **Br** 79.91	36 **Kr** 83.80
5	37 **Rb** 85.47	38 **Sr** 87.62	39 **Y** 88.91	40 **Zr** 91.22	41 **Nb** 92.91	42 **Mo** 95.94	43 **Tc** 98.91	44 **Ru** 101.07	45 **Rh** 102.91	46 **Pd** 106.4	47 **Ag** 107.87	48 **Cd** 112.40	49 **In** 114.82	50 **Sn** 118.69	51 **Sb** 121.75	52 **Te** 127.60	53 **I** 126.90	54 **Xe** 131.30
6	55 **Cs** 132.91	56 **Ba** 137.34	71 **Lu** 174.97	72 **Hf** 178.49	73 **Ta** 180.95	74 **W** 183.85	75 **Re** 186.2	76 **Os** 190.2	77 **Ir** 192.2	78 **Pt** 195.09	79 **Au** 196.97	80 **Hg** 200.59	81 **Tl** 204.37	82 **Pb** 207.19	83 **Bi** 208.98	84 **Po** 210	85 **At** 210	86 **Rn** 222
7	87 **Fr** 223	88 **Ra** 226.03	103 **Lr** 262.1	104 **Rf**	105 **Db**	106 **Sg**	107 **Bh**	108 **Hs**	109 **Mt**	110 **Uun**	111 **Uuu**	112 **Uub**	113 **Uut**					

← **Metals** | **Metalloids** | **Nonmetals** →

57 **La** 138.91	58 **Ce** 140.12	59 **Pr** 140.91	60 **Nd** 144.24	61 **Pm** 146.92	62 **Sm** 150.35	63 **Eu** 151.96	64 **Gd** 157.25	65 **Tb** 158.92	66 **Dy** 162.50	67 **Ho** 164.93	68 **Er** 167.26	69 **Tm** 168.93	70 **Yb** 173.04	Lanthanides
89 **Ac** 227.03	90 **Th** 232.04	91 **Pa** 231.04	92 **U** 238.03	93 **Np** 237.05	94 **Pu** 239.05	95 **Am** 241.06	96 **Cm** 247.07	97 **Bk** 249.08	98 **Cf** 251.08	99 **Es** 254.09	100 **Fm** 257.10	101 **Md** 258.10	102 **No** 255	Actinides

*Molar masses quoted to the number of significant figures given here can be regarded as typical of most naturally occurring samples.

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CONTENTS

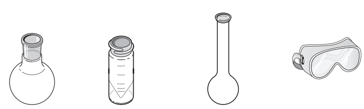
PREFACE

In preparing this Fourth Edition of *Laboratory Techniques in Organic Chemistry*, we have maintained our emphasis on the fundamental techniques that students encounter in the organic chemistry laboratory. We have also expanded our emphasis on the critical-thinking skills that students need to successfully carry out inquiry-driven experiments. The use of guided-inquiry and design-based experiments and projects is arguably the most important recent development in the teaching of the undergraduate organic chemistry lab, and it provides the most value added for our students.

Organic chemistry is an experimental science, and students learn its process in the laboratory. Our primary goal should be to teach students how to carry out well-designed experiments and draw reasonable conclusions from their results—a process at the heart of science. We should work to find opportunities that engage students in addressing questions whose answers come from their experiments, in an environment where they can succeed. These opportunities should be designed to catch students' interest, transforming them from passive spectators to active participants. A well-written and comprehensive textbook on the techniques of experimental organic chemistry is an important asset in reaching these goals.

Changes in the Fourth Edition

The Fourth Edition of *Laboratory Techniques in Organic Chemistry* builds on our strengths in basic lab techniques and spectroscopy, and includes a number of new features. To make it easier for students to locate the content relevant to their experiments, icons distinguish the techniques specific to each of the three common types of lab glassware—miniscale standard taper, microscale standard taper, and Williamson glassware—and also highlight safety concerns.



Sections on microwave reactors, flash chromatography, green chemistry, handling air-sensitive reagents, and measurement uncertainty and error analysis have been added or updated. The newly added Part 6 emphasizes the skills students need to carry out inquiry-driven experiments, especially designing and carrying out experiments based on literature sources. Many sections concerning basic techniques have been modified and reorganized to better meet the practical needs of students as they encounter laboratory work. Additional questions have also been added to a number of chapters to help solidify students' understanding of the techniques.

Short essays provide context for each of the six major parts of the Fourth Edition, on topics from the role of the laboratory to the spectrometric revolution. The essay "Intermolecular Forces in Organic Chemistry" provides the basis for subsequent discussions on organic separation and purification techniques, and the essay "Inquiry-Driven Lab Experiments" sets the stage for using guided-inquiry and design-based experiments. Rewritten sections on sources of confusion and common pitfalls help students avoid and solve technical problems that could easily discourage them if they did not have this practical support. We believe that these features provide an effective learning tool for students of organic chemistry.

Who Should Use This Book?

The book is intended to serve as a laboratory textbook of experimental techniques for all students of organic chemistry. It can be used in conjunction with any lab experiments to provide the background information necessary for developing and mastering the skills required for organic chemistry lab work. *Laboratory Techniques in Organic Chemistry* offers a great deal of flexibility. It can be used in any organic laboratory with any glassware. The basic techniques for using miniscale standard taper glassware as well as microscale 14/10 standard taper or Williamson glassware are all covered. The miniscale glassware that is described is appropriate with virtually any 14/20 or 19/22 standard taper glassware kit.

Modern Instrumentation

Instrumental methods play a crucial role in supporting modern experiments, which provide the active learning opportunities instructors seek for their students. We feature instrumental methods that offer quick, reliable, quantitative data. NMR spectroscopy and gas chromatography are particularly important. Our emphasis is on how to acquire good data and how to read spectra efficiently, with real understanding. Chapters on ^{1}H and ^{13}C NMR, IR, and mass spectrometry stress the practical interpretation of spectra and how they can be used to answer questions posed in an experimental context. They describe how to deal with real laboratory samples and include case studies of analyzed spectra.

Organization

The book is divided into six parts:

- Part 1 has chapters on safety, green chemistry, and the lab notebook.
- Part 2 discusses lab glassware, measurements, heating and cooling methods, setting up organic reactions, and computational chemistry.
- Part 3 introduces filtration, extraction, drying organic liquids and recovering products, distillation, refractometry, melting points, recrystallization, and the measurement of optical activity.
- Part 4 presents the three chromatographic techniques widely used in the organic laboratory—thin-layer, liquid, and gas chromatography.
- Part 5 discusses IR, ^{1}H and ^{13}C NMR, MS, and UV-VIS spectra in some detail.
- Part 6 introduces the design and workup of chemical reactions based on procedures in the literature of organic chemistry.

Traditional organic qualitative analysis is available on our Web site: www.whfreeman.com/mohrig4e.

Modern Projects and Experiments in Organic Chemistry

The accompanying laboratory manual, *Modern Projects and Experiments in Organic Chemistry,* comes in two complete versions:

- *Modern Projects and Experiments in Organic Chemistry: Miniscale and Standard Taper Microscale* (ISBN 0-7167-9779-8)
- *Modern Projects and Experiments in Organic Chemistry: Miniscale and Williamson Microscale* (ISBN 0-7167-3921-6)

Modern Projects and Experiments is a combination of inquiry-based and traditional experiments, plus multiweek inquiry-based projects. It is designed to provide quality content, student accessibility, and instructor flexibility. This laboratory manual introduces students to the way the contemporary organic lab actually functions and allows them to experience the process of science. All of its experiments and projects are also available through LabPartner Chemistry.

LabPartner Chemistry

W. H. Freeman's latest offering in custom lab manuals provides instructors with a diverse and extensive database of experiments published by W. H. Freeman and Hayden-McNeil Publishing—all in an easy-to-use, searchable online system. With the click of a button, instructors can choose from a variety of traditional and inquiry-based labs, including the experiments from *Modern Projects and Experiments in Organic Chemistry*. LabPartner Chemistry sorts labs in a number of ways, from topic, title, and author, to page count, estimated completion time, and prerequisite knowledge level. Add content on lab techniques and safety, reorder the labs to fit your syllabus, and include your original experiments with ease. Wrap it all up in an array of bindings, formats, and designs. It's the next step in custom lab publishing. Visit http://www.whfreeman.com/labpartner to learn more.

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copyediting and publication processes. We thank Diana Blume for her creative design elements. Finally, we express heartfelt thanks for the patience and support of our spouses, Adrienne Mohrig, Ellie Schatz, and Bill Hammond, during the several editions of *Laboratory Techniques in Organic Chemistry*.

We hope that teachers and students of organic chemistry find our approach to laboratory techniques effective, and we would be pleased to hear from those who use our book. Please write to us in care of the Chemistry Acquisitions Editor at W. H. Freeman and Company, 41 Madison Avenue, New York, NY 10010, or e-mail us at chemistry@whfreeman.com.

PART

1

Introduction to the Organic Laboratory

Essay—The Role of the Laboratory

Organic chemistry provides us with a framework to understand ourselves and the world in which we live. Organic compounds are present everywhere in our lives—they comprise the food, fabrics, cosmetics, and medications that we use on a daily basis. By studying how the molecules of life interact with one another, we can understand the chemical processes that sustain life and discover new compounds that could potentially transform our lives. For example, organic chemistry was used to discover the cholesterol-lowering blockbuster drug, Lipitor®. Current research in organic semiconductors, which are more flexible, cheaper, and lighter in weight than silicon-based components, could lead to solar cells incorporated into clothing, backpacks, and virtually anything. The purpose of this textbook is to provide *you* with the skills and knowledge to make new discoveries like these, view the world from a new perspective, and ultimately harness the power of organic chemistry.

It is in the laboratory that we learn "how we know what we know." The lab deals with the processes of scientific inquiry that organic chemists use. Although the techniques may at first appear complicated and mysterious, they are essential tools for addressing the central questions of this experimental science, which include:

- What chemical compounds are present in this material?
- What is this compound and what are its properties?
- Is this compound pure?
- How could I make this compound?
- How does this reaction take place?
- How can I separate my product from other reaction side products?

Keep in mind that the skills you will be learning are very practical and there is a reason for each and every step. You should make it your business to understand **why** these steps are necessary and **how** they accomplish the desired result. If you can answer these questions for every lab session, you have fulfilled the most basic criterion for satisfactory lab work.

You may also have opportunities to test your own ideas by designing new experiments. Whenever you venture into the unknown, it becomes even more important to be well informed and organized *before you start any experiments.* Safety should be a primary concern, so you will need to recognize potential hazards, anticipate possible outcomes, and responsibly dispose of chemical waste. In order to make sense of your data and report your findings to others, you will need to keep careful records of your experiments. The first section of this textbook introduces you to reliable sources of information, safety procedures, ways to protect the environment, and standards for laboratory record-keeping. It is important to make these practices part of your normal laboratory routine. If you are ever unsure about your preparation for lab, ask your instructor.

There is no substitute for witnessing chemical transformations and performing separation processes in the laboratory. Lab work enlivens the chemistry that you are learning "on paper" and helps you understand how things work. Color changes, phase changes, and spectral data are fun to witness and fun to analyze and understand. Enjoy this opportunity to experiment in chemistry and come to lab prepared and with your brain engaged!

CHAPTER

1

SAFETY IN THE LABORATORY

All of the stories in this chapter are based on the authors' experiences working and teaching in the lab.

Carrie used a graduated cylinder to measure a volume of concentrated acid solution at her lab bench. As she prepared to record data in her notebook later in the day, she picked up her pen from the bench-top and absent-mindedly started chewing on the cap. Suddenly, she felt a burning sensation in her mouth and yelled, "It's hot!" The lab instructor directed her to the sink to thoroughly rinse her mouth with water and she suffered no long-term injury.

This incident is like most laboratory accidents; it resulted from inappropriate lab practices and inattention, and it was preventable. Carrie should have handled the concentrated acid in a fume hood and, with advice from her instructor, immediately cleaned up the acid she must have spilled. She should never have introduced any object in the lab into her mouth. With appropriate knowledge, most accidents are easily remedied. In this case, the instructor knew from her shout what the exposure must have been and advocated a reasonable treatment.

Accidents in teaching laboratories are extremely rare; instructors with 20 years of teaching experience may witness fewer than five mishaps. Instructors and institutions continually implement changes to the curriculum and laboratory environment that improve safety. Experiments are now designed to use very small amounts of material, which minimizes the hazards associated with chemical exposure and fire. Laboratories provide greater access to fume hoods for performing reactions, and instructors choose the least hazardous materials for accomplishing transformations. Nevertheless, you play an important role in ensuring that the laboratory is as safe as possible.

You can rely on this textbook and your teacher for instruction in safe and proper laboratory procedures. **You are responsible for developing good laboratory habits**: Know and understand the laboratory procedure and associated hazards, practice good technique, and be aware of your actions and the actions of those around you. Habits like these are transferable to other situations and developing them will not only enable you to be effective in the laboratory but also help you to become a valuable employee and citizen.

The goal of safety training is to manage hazards in order to minimize the risk of accidental chemical exposure, personal injury, or damage to property or the environment.

- **Before you begin laboratory work,** familiarize yourself with the general laboratory safety rules (listed below) that govern work at any institution.
- **At the first meeting of your lab class,** learn institutional safety policies regarding personal protective equipment (PPE), the location and use of safety equipment, and procedures to be followed in emergency situations.
- **For each individual experiment,** note the safety considerations identified in the description of the procedure, the hazards

associated with the specific chemicals you will use, and the waste disposal instructions.

In addition to knowledge of basic laboratory safety, you need to learn how to work safely with organic chemicals. Many organic compounds are flammable or toxic. Many can be absorbed through the skin; others are volatile and can be ingested by inhalation. Become familiar with and use chemical hazard documentation, such as the Globally Harmonized System (GHS) of hazard information and Material Safety Data Sheets (MSDSs) or Safety Data Sheets (SDSs). Despite the hazards, organic compounds can be handled with a minimum of risk if you are adequately informed about the hazards and safe handling procedures, and if you use common sense while you are in the lab.

1.1 General Safety Information

General Safety Rules

1. **Do not work alone in the laboratory.** Being alone in a situation in which accidents can occur can be life threatening.
2. **Always perform an experiment as specified.** Do not modify the conditions or perform new experiments without authorization from your instructor.
3. **Wear clothing that covers and protects your body; use appropriate protective equipment, such as goggles and gloves; and tie back long hair at all times in the laboratory.** Shorts, tank tops, bare feet, sandals, or high heels are not suitable attire for the lab. Loose clothing and loose long hair are fire hazards or could become entangled in an apparatus. Wear safety glasses or chemical splash goggles at all times in the laboratory. Laboratory aprons or coats may be required by your instructor.
4. **Be aware of others working near you** and the hazards associated with their experiments. Often the person hurt worst in an accident is the one standing next to the place where the accident occurred. Communicate with others and make them aware of the hazards associated with your work.
5. **Never eat, drink, chew gum, apply makeup, or remove or insert contact lenses in the laboratory. Never directly inhale or taste any substance or introduce any laboratory equipment, such as a piece of glassware or a writing utensil, into your mouth.** Wash your hands with soap and water before you leave the laboratory to avoid accidentally contaminating the outside environment, including items that you may place into your mouth with your hands.
6. **Notify your instructor if you have chemical sensitivities or allergies or if you are pregnant.** Discuss these conditions and the advisability of working in the organic chemistry laboratory with appropriate medical professionals.
7. **Read and understand the hazard documentation regarding any chemicals you plan to use in an experiment.** This can be found in Material Safety Data Sheets (MSDSs) or Safety Data Sheets (SDSs).

8. **Know where to find and how to use safety equipment,** such as the eye wash station, safety shower, fire extinguisher, fire blanket, first aid kit, telephone, and fire alarm pulls.
9. **Report injuries, accidents, and other incidents to your instructor** and follow his or her instructions for treatment and documentation.
10. **Properly dispose of chemical waste**, including chemically contaminated disposable materials, such as syringes, pipets, gloves, and paper. **Do not dispose of any chemicals by pouring them down the drain or putting them in the trash can without approval from your instructor.**

Chemical Hygiene Plan

Your institution will have a chemical hygiene plan that outlines the safety regulations and procedures that apply in your laboratory. It will provide contact information and other information about local safety rules and processes for managing laboratory fires, injuries, chemical spills, and chemical waste. You can search the institutional web pages or ask your instructor for access to the chemical hygiene plan.

1.2 Preventing Chemical Exposure

Mary was wearing nitrile gloves while performing an extraction with dichloromethane. Although she spilled some solution on her gloves, she continued working until she felt her hands burning. She peeled off the gloves and washed her hands thoroughly, but a burning sensation under her ring persisted for 5 to 10 minutes thereafter. She realized that the dichloromethane solution easily passed through her gloves and she wondered whether her exposure to dichloromethane and the compounds dissolved in it would have an adverse effect on her health.

Personal Protective Equipment

This example illustrates the importance of understanding the level of protection provided by ***personal protective equipment (PPE)*** and other safety features in the laboratory.

- **Never assume that clothing, gloves, lab coats, or aprons will protect you from every kind of chemical exposure.** If chemicals are splashed onto your clothing or your gloves, remove the articles immediately and thoroughly wash the affected area of your body.
- If you spill a chemical directly on your skin, wash the affected area thoroughly with water for 10–15 min, and notify your instructor.

Eye protection. Safety glasses with side shields have impact-resistant lenses that protect your eyes from flying particles, but they provide little protection from chemicals. Chemical splash goggles fit snugly against your face and will guard against the impact from flying objects *and* protect your eyes from liquid splashes, chemical vapors, and particulate or corrosive chemicals. These are the best choice for the organic chemistry laboratory and your instructor will be able to recommend an appropriate style to purchase. If you wear prescription eyeglasses, you should wear chemical splash goggles over your corrective lenses. Contact lenses could be damaged from exposure to chemicals and therefore you should not wear them in the laboratory. Nevertheless, many organizations have removed restrictions on wearing contact lenses in the lab because concerns that they contribute to the likelihood or severity of eye damage seem to be unfounded. If you choose to wear contact lenses in the laboratory, you must also wear chemical splash goggles to protect your eyes. Because wearing chemical splash goggles is one of the most important steps you can take to safely work in the laboratory, **we will use a splash goggle icon (see margin figure) to identify important safety information throughout this textbook**.

Protective attire. Clothes should cover your body from your neck to at least your knees and shoes should completely cover your feet in the laboratory. Cotton clothing is best because synthetic fabrics could melt in a fire or undergo a reaction that causes the fabric to adhere to the skin and severely burn it. Wearing a lab coat or apron will help protect your body. For footwear, leather provides better protection than other fabrics against accidental chemical spills. Your institution may have more stringent requirements for covering your body.

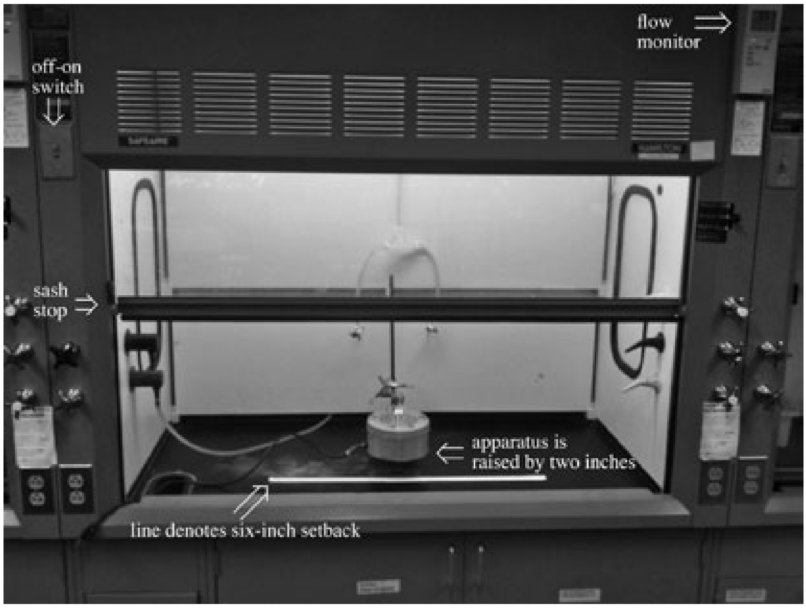
Disposable gloves. Apart from goggles, gloves are the most common form of PPE used in the organic laboratory. Because disposable gloves are thin, many organic compounds permeate them quickly and they provide "splash protection" only. **This means that once you spill chemicals on your gloves, you should remove them, wash your hands thoroughly, and put on a fresh pair of gloves.** Ask your instructor how to best dispose of contaminated gloves.

Table 1.1 lists a few common chemicals and the chemical resistance to each one provided by three common types of gloves. A

TABLE 1.1 Chemical resistance of common types of gloves to various compounds

	Glove type		
Compound	**Neoprene**	**Nitrile**	**Latex**
Acetone	Good	Fair	Good
Chloroform	Good	Poor	Poor
Dichloromethane	Fair	Poor	Poor
Diethyl ether	Very good	Good	Poor
Ethanol	Very good	Good	Good
Ethyl acetate	Good	Poor	Fair
Hexane	Excellent	Excellent	Poor
Hydrogen peroxide	Excellent	Good	Good
Methanol	Very good	Fair	Fair
Nitric acid (conc.)	Good	Poor	Poor
Sodium hydroxide	Very good	Good	Excellent
Sulfuric acid (conc.)	Good	Poor	Poor
Toluene	Fair	Fair	Poor

The information in this table was compiled from http://www.microflex.com, http://www.ansellpro.com, and "Chemical Resistance and Barrier Guide for Nitrile and Natural Rubber Latex Gloves," Safeskin Corporation, San Diego, CA, 1999.



Gretchen Hofmeister

FIGURE 1.1 A typical chemical fume hood.

more extensive chemical resistance table for types of gloves may be posted in your laboratory. Additional information on disposable gloves and tables listing glove types and their chemical resistance are also available from many websites, for example:

http://www.microflex.com
http://www.ansellpro.com
http://chemistry.umeche.maine.edu/Safety.html

Chemical Fume Hoods

You can protect yourself from accidentally inhaling noxious chemical fumes, toxic vapors, or dust from finely powdered materials by handling chemicals inside a fume hood. A typical fume hood with a movable sash is depicted in Figure 1.1. The sash is constructed of laminated safety glass and can open and close either vertically or horizontally. When the hood is turned on, a continuous flow of air sweeps over the bench top and removes vapors or fumes from the area. The volume of air that flows through the sash opening is constant, so the rate of flow, or face velocity, is greater when the sash is closed than when it is open. Most hoods have stops or signs indicating the maximum open sash position that is safe for handling chemicals. If you are unsure what is a safe sash position for the hoods in your laboratory, ask your instructor.

Because many compounds used in the organic laboratory are at least potentially dangerous, the best practice is to run every experiment in a hood, if possible. Your instructor will tell you when an experiment *must* be carried out in a hood.

- **Make sure that the hood is turned on before you use it.**
- Never position your face near the sash opening or place your head inside a hood when chemicals are present. Keep the sash in front of your face so that you look through the sash to monitor what is inside the hood.
- Place chemicals and equipment at least six inches behind the sash opening.

- Elevate reaction flasks and other equipment at least two inches above the hood floor to ensure good airflow around the apparatus.
- When you are not actively manipulating equipment in the hood, adjust the sash so that it covers most of the hood opening and shields you from the materials inside.

A link to a YouTube video, created at Dartmouth College, which describes the function and use of fume hoods, can be found at: http://www.youtube.com/watch?v=nlAaEpWQdwA .

Chemical Hygiene

Poor housekeeping often leads to accidental chemical exposure. In addition to your own bench area, the balance and chemical dispensing and waste areas must be kept clean and orderly.

- If you spill anything while measuring out your chemicals, notify your instructor and clean it up immediately.
- After weighing a chemical, replace the cap on the container and dispose of the weighing paper in the appropriate receptacle.
- Clean glassware, spatulas, and other equipment as soon as possible after using them.
- Always remove gloves, lab coat, or apron before leaving the laboratory to prevent widespread chemical contamination.
- Dispose of chemical waste appropriately.

1.3 Preventing Cuts and Burns

As Harvey adjusted a pipet bulb over the end of a disposable glass pipet, the pipet broke and the broken end jammed into his thumb, cutting it badly. Harvey required hand surgery to repair a damaged nerve and he could not manipulate his thumb for several months afterward.

Cuts

While Harvey's accident was unusually severe, the most common laboratory injuries are cuts from broken glass or puncture wounds from syringe needles. For this reason, handle glassware and sharp objects with care.

- Check the rims of beakers, flasks, and other glassware for chips and discard any piece of glassware that is chipped.
- If you break a piece of glassware, use a dustpan and broom instead of your hands to pick up the broken pieces.
- Do not put broken glass or used syringe needles in the trash can. Dispose of them separately—broken glass in the broken glass container and syringe needles in the sharps receptacle.
- If a stopper, stopcock, or other glass item seems stuck, do not force it. Ask your instructor, who is more experienced with the equipment, for assistance in these cases.
- To safely insert thermometers or glass tubes into corks, rubber stoppers, and thermometer adapters, lubricate the end of the glass with a drop of water or glycerol, hold the tube near the lubricated end, and insert it slowly by gently rotating it.

- **Never push on the end of a glass tube or a thermometer to insert it into a stopper; it may break and the shattered end could be driven into your hand.**

Burns

Remember that glass and the tops of hot plates look the same when they are hot as when they are cold. Steam and hot liquids also cause severe burns. Liquid nitrogen and dry ice can quickly give you frostbite.

- Do not put hot glass on a bench where someone else might pick it up.
- Turn off the steam source before removing containers from the top of a steam bath.
- The screws or valve stems attached to the rounded handle that controls a steam line can become very hot; be careful not to touch them when you turn the steam on or off.
- Move containers of hot liquids only if necessary and use a clamp, tongs, rubber mitts, or oven gloves to hold them.
- Wear insulated gloves when handling dry ice and wear insulated gloves, a face shield, long pants, and long sleeves when dispensing liquid nitrogen.

1.4 Preventing Fires and Explosions

Michael was purifying a reaction product by distillation on the laboratory bench. The product mixture also contained diethyl ether. About halfway through the distillation, the distilled material caught fire. Michael's instructor used a fire extinguisher to put out the fire and assisted Michael in turning off the heating mantle and lifting the distillation system away from the heat source. As soon as possible, the entire apparatus was relocated to the fume hood and Michael was instructed to chill the receiving flask in an ice bath, to minimize the escape of flammable vapors from the flask.

Fires

Hydrocarbons and many of their derivatives are flammable and the potential for fire in the organic laboratory always exists. Fortunately, most modern lab procedures require only small amounts of material, minimizing the risk of fire. ***Flammable*** compounds do not spontaneously combust in air; they require a spark, a flame, or heat to catalyze the reaction. Vapors from low-boiling organic liquids, such as diethyl ether or pentane, can travel over long distances at bench or floor level (they are heavier than air) and thus they are susceptible to ignition by a source that is located up to 10 ft away. The best way to prevent a fire is to prevent ignition.

Four sources of ignition are present in the organic laboratory: **open flames**, **hot surfaces** such as hot plates or heating mantles (Figure 1.2), **faulty electrical equipment**, and **chemicals**. Flames, such as those produced by Bunsen burners, should be used rarely in the organic laboratory and only with the permission of your instructor. Hot plates and heating mantles, however, are used routinely. The thermostat on most hot plates is not sealed and can spark when it cycles on and off. The spark can ignite flammable vapors from