

ESSENTIALS OF ORGANIC CHEMISTRY

Small Scale Laboratory Experiments



Murray Zanger
James McKee

Essentials of Organic Chemistry

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James R. McKee Ph.D.

Murray Zanger Ph.D.

Philadelphia College of Pharmacy and Sciences



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Cover Design *Shepherd, Inc.*
Production, Design, and Composition *Shepherd, Inc.*

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Library of Congress Catalog Card Number 96-78880

ISBN 0-697-33009-5

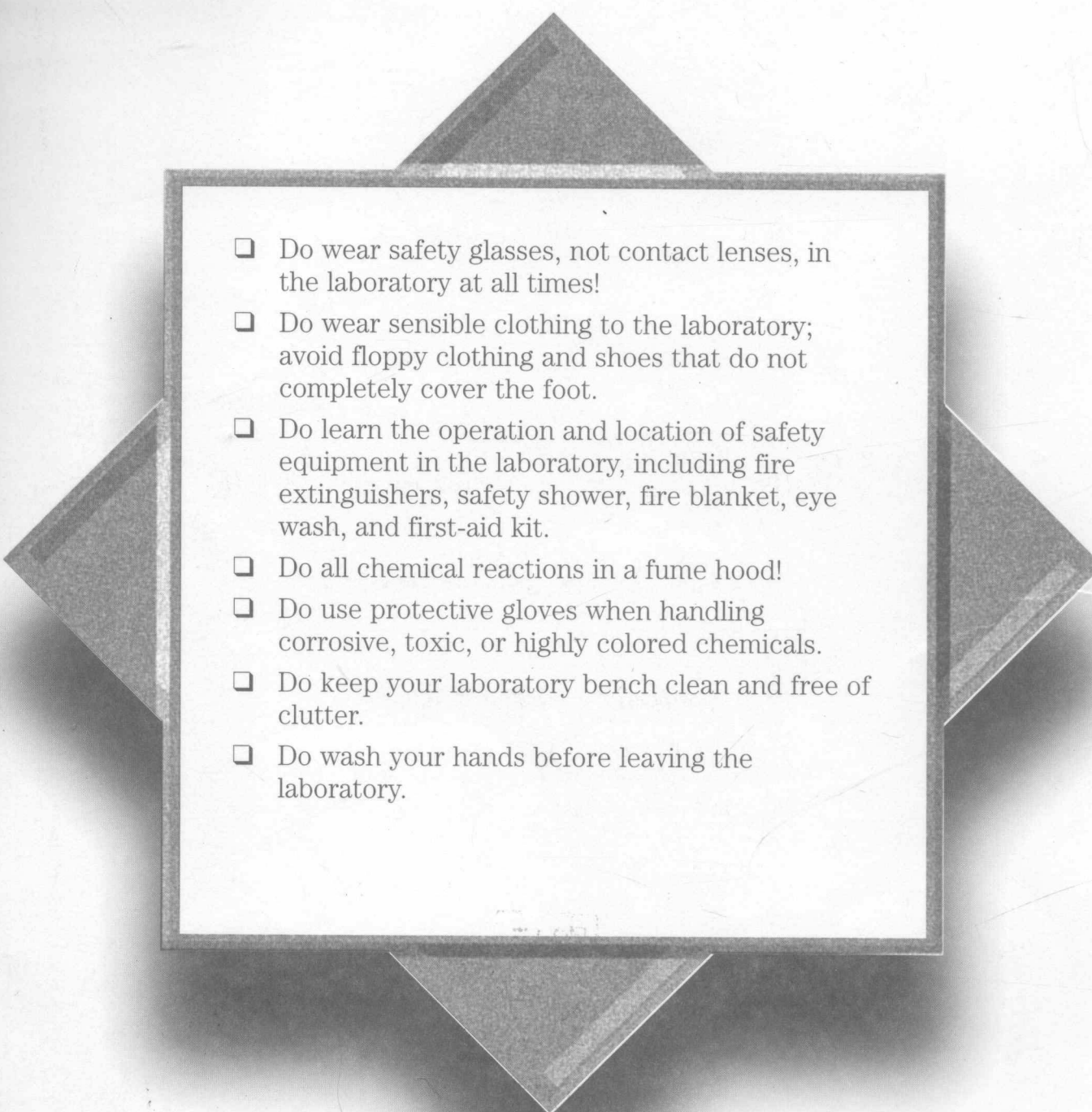
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Some of the laboratory experiments included in this text may be hazardous if materials are handled improperly or if procedures are conducted incorrectly. Safety precautions are necessary when you are working with chemicals, glass test tubes, hot water baths, sharp instruments, and the like, or for any procedures that generally require caution. Your school may have set regulations regarding safety procedures that your instructor will explain to you. Should you have any problems with materials or procedures, please ask your instructor for help.

Printed in the United States of America
2460 Kerper Boulevard, Dubuque, IA 52001

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Laboratory Safety Do's

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- ☐ Do wear safety glasses, not contact lenses, in the laboratory at all times!
 - ☐ Do wear sensible clothing to the laboratory; avoid floppy clothing and shoes that do not completely cover the foot.
 - ☐ Do learn the operation and location of safety equipment in the laboratory, including fire extinguishers, safety shower, fire blanket, eye wash, and first-aid kit.
 - ☐ Do all chemical reactions in a fume hood!
 - ☐ Do use protective gloves when handling corrosive, toxic, or highly colored chemicals.
 - ☐ Do keep your laboratory bench clean and free of clutter.
 - ☐ Do wash your hands before leaving the laboratory.

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To our wives, Marcia and Trudy, whose encouragement and support and enormous patience kept us at our task.

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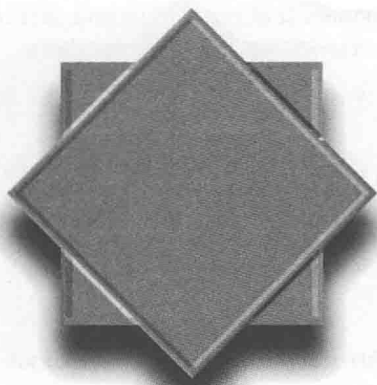
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Preface

Why have an organic chemistry laboratory for non-chemistry students? This question will no doubt be asked by many, especially those students who are required to take organic chemistry. Arguably, it can be stated that “educated people” in our modern, scientifically and technologically oriented society cannot make that claim unless they have some knowledge about the complex world we live in. How does a gasoline engine work? What principles were used for the creation of TV? Is “natural” vitamin C better than synthetic vitamin C? The ability to answer these and many other questions delineate between those who merely consume and those who can contribute to progress. Organic chemistry, a fundamental science, is an integral part of everyone’s life. The clothing we wear is made either from natural or synthetic fibers, both of which are organic compounds. The whole range of plastics, which both enrich and pollute our existence, are man-made organic substances. The dyes on our clothing, cosmetics, foodstuffs (natural, artificial, or hybrid mixtures) and pharmaceuticals are all organic creations of either an organic chemist or the Creator. Some knowledge then of organic chemistry can enable one to have a better understanding of the world we live in.

In this book, we have sought to create a text that supplements a one-semester lecture course in organic chemistry. Neither the lecture nor lab portions of the course are designed to produce a chemist. Rather the lectures and more especially the laboratory experience should engender an appreciation of what organic chemistry is, how it is practiced, and how it applies to the “real world.”

As a means of realizing the goals expressed above, the text consists of fourteen chapters, with the first two containing basic information about laboratory safety, waste disposal, instrumental methods, etc. The remaining twelve chapters consist of experiments that illustrate some basic organic chemical reactions that also relate to everyday materials.

One experiment involves the purification of aspirin that has partially decomposed. In another, some of the properties of sucrose (table sugar) are studied. Since polymers play an important part in all of our lives, students will have the opportunity to prepare several polymers; among these are plexiglass and nylon. Indigo, the dye used for blue jeans, is synthesized and used to dye a piece of cloth. For the weight conscious, we have included a synthesis of the artificial sweetener, saccharin. A somewhat exotic experiment has the student preparing rubrene peroxide, which, on decomposition, produces light.

Even though instruments may not be available for this type of course, one experiment will give the student an appreciation of what NMR can do in terms of structure determination. Other experiments utilize gas or thin-layer chromatography. The use of molecular models, the polarimeter, etc. are also included in this manual. In addition, there are several experiments where the student must identify or characterize an unknown compound. In summary, there are enough experiments covering a wide range of difficulty, and instrumental and chemical techniques to permit instructors to choose a set that will meet their particular interests and needs.

As a bridge between the chemist and the non-scientist, we have included a short introductory essay at the beginning of each experiment that relates specifically either to the experiment or to the chemistry illustrated. These essays attempt to make the science relevant to someone whose major areas of interest lie outside the realm of chemistry. Among these are a brief history of the evolution of Magnetic Resonance Imaging from

Nuclear Magnetic Resonance, a short discussion of the pros and cons of the polymers that are an integral part of all of our lives, the tragic story of thalidomide, the development of artificial sweeteners, and numerous others.

In all of the other experiments, the emphasis is placed on safety and the clarity of experimental directions. To achieve this the experimental write-up follows a novel format.

1. A brief description and rationale for the experiment including an equation for the reaction.
2. A three-column format consisting of
 - a. An icon column that visually depicts the equipment or technique being used in that step of the procedure. These figures reveal the "flow" of the experiment.
 - b. A series of short, numbered steps for the procedure.
 - c. A column that includes experimental notes, cautions, waste disposal instructions, and questions about the experiment.
3. A small but educational selection of IR spectra for those experiments where they might be useful for confirmation.
4. A series of questions designed to make the student think about why certain procedures were used.
5. Cross-references to organic textbooks where an expanded discussion of the organic reaction or technique is covered.

In short we have attempted to create a book that can be used at different levels and with a wide selection of experiments. We have tried to address many of the suggestions and criticisms of the reviewers but satisfying everyone can be self-defeating. From those who do adopt this book, we welcome comments and suggestions for ways to further refine our work. Writing a book of this type always seemed easy when we were reading and using other texts. The truth is that enormous effort is actually required. We hope users will find these efforts worthwhile.

We'd like to thank S. Todd Deal, Georgia Southern University, for his accuracy review of this text. Our thanks also go to the following reviewers:

Joanna H. Fribush
North Adams State College

Steven A. Hardinger
California State University, Fullerton

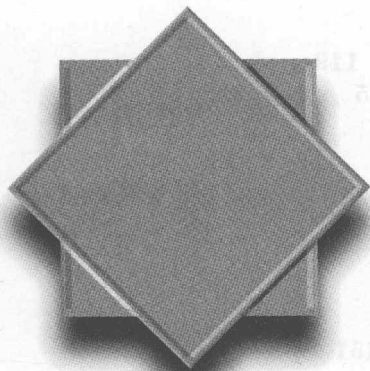
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CHAPTER 1

Introduction to the Laboratory

I. Laboratory Safety

- A. *Safety Equipment:* A set of safety rules is written on the inside front cover of this book. Careful observation of these rules will help to prevent accidents in the laboratory; however, from time to time accidents can occur. Therefore, safety equipment is installed for this eventuality in the laboratory. Safety equipment and practices should include the following:

Laboratory Safety

- An eye wash
- A safety shower
- Fire extinguishers
- Hoods
- First-aid kit

Personal Safety

- Chemical splash goggles
- Protective clothing
- No food or drink in the laboratory
- Personal hygiene

1. *Eye Wash:* The eye wash is designed to flush irritating chemicals from the eyes. It should be capable of providing a stream of water for at least 15 minutes. In the event of an eye accident, you should proceed to the eye wash at once and wash the eye for at least 15 minutes. During this process, the eye should be kept open. The eyes are the most vulnerable part of the body. In the event of any eye injury, notify the instructor at once. All eye injuries should be examined immediately by a health professional.

Never use the eye wash for anything other than its intended purpose.

2. *Safety Shower:* The safety shower is designed for two purposes; namely, to extinguish clothing fires and to provide a whole body wash if a large chemical spill occurs.
 - a. *Clothing Fires:* If your clothing catches fire, the best rule is to fall and roll. Never run to a shower with your clothes on fire; it will only fan the flames. Use the shower afterwards to squelch any residual embers.
 - b. *Large Chemical Spills:* Large chemical spills on clothing or exposed parts of the body should be removed at once using the safety shower. Contaminated clothing should be removed, and the affected body areas should be thoroughly washed to remove any chemical traces. Do not reuse contaminated clothing until it has been completely washed! Serious and avoidable injuries have resulted from wearing contaminated clothing.
3. *Fire Extinguishers:* In the laboratory, you will sometimes work with flammable materials. ABC fire extinguishers are adequate for extinguishing most fires. Several of these extinguishers should be

placed in the laboratory. Learn their locations. Your instructor will demonstrate their use before you begin to work in the laboratory.

4. *Hoods*: If possible, do all experiments in a hood. The ventilation system draws the fumes generated by an experiment away from the experimenter. The walls of the hood enclose the experiment on five sides. Therefore, if an explosion or spill occurs, the experiment can be contained. A sliding transparent sash augments all of these features. The sash should always be kept between the individual's eyes and the experiment. In a modern organic laboratory, chemical reactions are always done in a hood.
5. *First-Aid Kits*: First-aid kits are used to treat minor injuries. Report all cuts and burns to the instructor and, at his or her discretion, visit the school physician for further treatment.
 - a. *Cuts*: All cuts should be cleaned carefully to remove any chemical residue or broken glass before a Band-Aid is applied.
 - b. *Burns*: Immediately flush burns under cold water for 15–20 minutes to reduce the magnitude of the injury. Do not rub the affected area or pack it in ice.

If a seemingly minor injury appears to be getting worse, consult a physician.

B. *Personal Protective Equipment*: Wearing the proper clothing during an experiment is as important to an individual's safety as is any other safety feature of the laboratory. This protective clothing should include the following items.

1. *Safety Glasses*: Safety glasses or goggles must be worn from the time you enter the laboratory until you leave the laboratory. There are *no exceptions* to this regulation! These glasses must be OSHA approved (Z 87.21).

Do not wear contact lenses in the laboratory. The lenses may actually trap organic vapors and hold them against the eye. Additionally, in the event of a splash, the material can be drawn under the contact lens by capillary action. Serious injury can result.

Ordinary glasses are *not* safety glasses. In the event of a splash, they do not provide lateral protection. In addition, street glasses are frequently made of plastic, and they can be ruined easily by the solvents in the laboratory.

2. *Lab Coats*: Lab coats are designed to be removed quickly in case of a fire or chemical spill. Lab coats provide protection against the minor spills and splashes of the laboratory. The coat should at least protect the upper body from the neck to the waist, and preferably, it should protect it to the knees. In addition, long pants should be worn to protect the lower body from spills and splashes.

The lab coat should be made of cotton, not synthetic materials. During a clothing fire, a synthetic material melts and becomes incorporated into the burn. Synthetic lab coats also tend to dissolve in organic solvents; therefore, they are not as durable as cotton ones.

3. *Shoes*: Proper laboratory footwear completely covers the foot. It may be either a street shoe or a sneaker, but sandals or open-toed shoes should not be worn. It is advisable to have a pair of sneakers available and to change into them before the lab period begins.
4. *Gloves*: If toxic or colored (dyes) substances are used in the laboratory, the instructor may advise wearing gloves. Gloves should be removed just prior to leaving the work area, otherwise contamination from the gloves may be transferred to otherwise clean environments (equipment, rooms, telephones, or other individuals).

Disposable gloves are preferred, and they should be worn only for as long as necessary.

C. *Good Laboratory Practice*

1. *Food or Drink in the Laboratory*: Food and drink should not be brought into the laboratory. Packaged materials (including lunches) can absorb materials from the air or bench top. Food consumed in the laboratory can easily become contaminated. Also, beverages can easily absorb toxic vapors from the air. Serious cases of poisoning have resulted from this type of occurrence.
2. *Smoking*: Cigarette smoking is banned from the laboratory for the following two important reasons:

- a. As with food consumption, materials from the air, hands, and desk can be carried to the mouth during smoking.
 - b. Cigarettes represent an unacceptable ignition hazard in the laboratory.
3. *Cleanliness*: During a laboratory experiment, you are exposed to a wide variety of chemicals. They can be retained on the hands, especially under the fingernails. It is a good laboratory practice to make sure your hands are clean before you leave the laboratory. Additionally, every effort should be made to prevent contamination of items brought into the laboratory (e.g., pencils, pens, and books). These items can act as a secondary source of exposure to chemicals (e.g., placing a contaminated pen in your mouth). Care should be exercised to avoid this serious hazard.

D. *Safe Handling of Laboratory Equipment*

1. *Heat Sources*: Heating mantles and steam baths are used as heat sources in the laboratory. Each source has its appropriate uses and precautions.
 - a. *Heating Mantles*: Heating mantles use electricity as a source of heat; therefore, they should be kept dry. When used, they should be connected to power only through a ground-fault interrupter.
 - b. *Steam Baths*: Steam baths provide a convenient source of heat for temperatures ranging from room temperature to 95°C. Steam, however, has a high heat of vaporization, and can cause severe scalding.
2. *Electrical Equipment*: Electrical equipment represents two significant hazards in the organic laboratory.
 - a. *Ignition Hazard*: Electrical motors spark frequently during operation. These sparks can cause fires or explosions. For this reason, in areas where solvents are located only spark-free motors should be used. The problem of sparking also exists with switches and plug connections. These devices should be on the outside of the hood where the concentration of solvent vapor is low and where the danger of igniting it is minimal.
 - b. *Shock Hazard*: Do not use poorly maintained equipment (e.g., frayed cords and loose plugs). Keep these devices dry and away from the puddles of water that may collect in a hood. All these devices should be connected through ground-fault interrupters to minimize shock hazards.
3. *Glassware*: In any laboratory experiment, it is essential that the correct equipment be used; moreover, many experiments have been ruined by using the wrong, defective, or improperly maintained equipment. This section will describe the proper use and care of common types of glassware found in the organic laboratory.
 - a. *Standard Taper Glassware*: The equipment in Figure 1.1 is equipped with a ground glass fitting (Standard Taper) that is usually $\text{T}14/20$, $\text{T}19/22$, or $\text{T}24/40$. Any male $\text{T}19/22$ joint will fit a female $\text{T}19/22$ joint.

Condensers, distillation heads, Claisen adapters, vacuum adapters, separatory funnels, and round-bottomed flasks are commonly fitted with these joints.

These pieces of equipment can be assembled into a variety of configurations for doing a chemical reaction, refluxing a solution, or purifying a mixture through distillation.

- i. *Storage*: Standard taper glassware can be cracked or broken if it is placed improperly in a drawer. Usually this type of glassware is stored in a case specifically designed for it. If such a storage case is not available, the glassware should be stored carefully to prevent it from breaking. The drawer should be opened and closed gently.
- ii. *Uses*: Before use, the tapered joints of the glassware items should be inspected for chips and cracks that run through the taper and give a poor seal when the apparatus is assembled. All portions of the glassware should be checked carefully for cracks (especially star cracks). These flaws expand as the container is heated, which causes the apparatus to fail and ruin

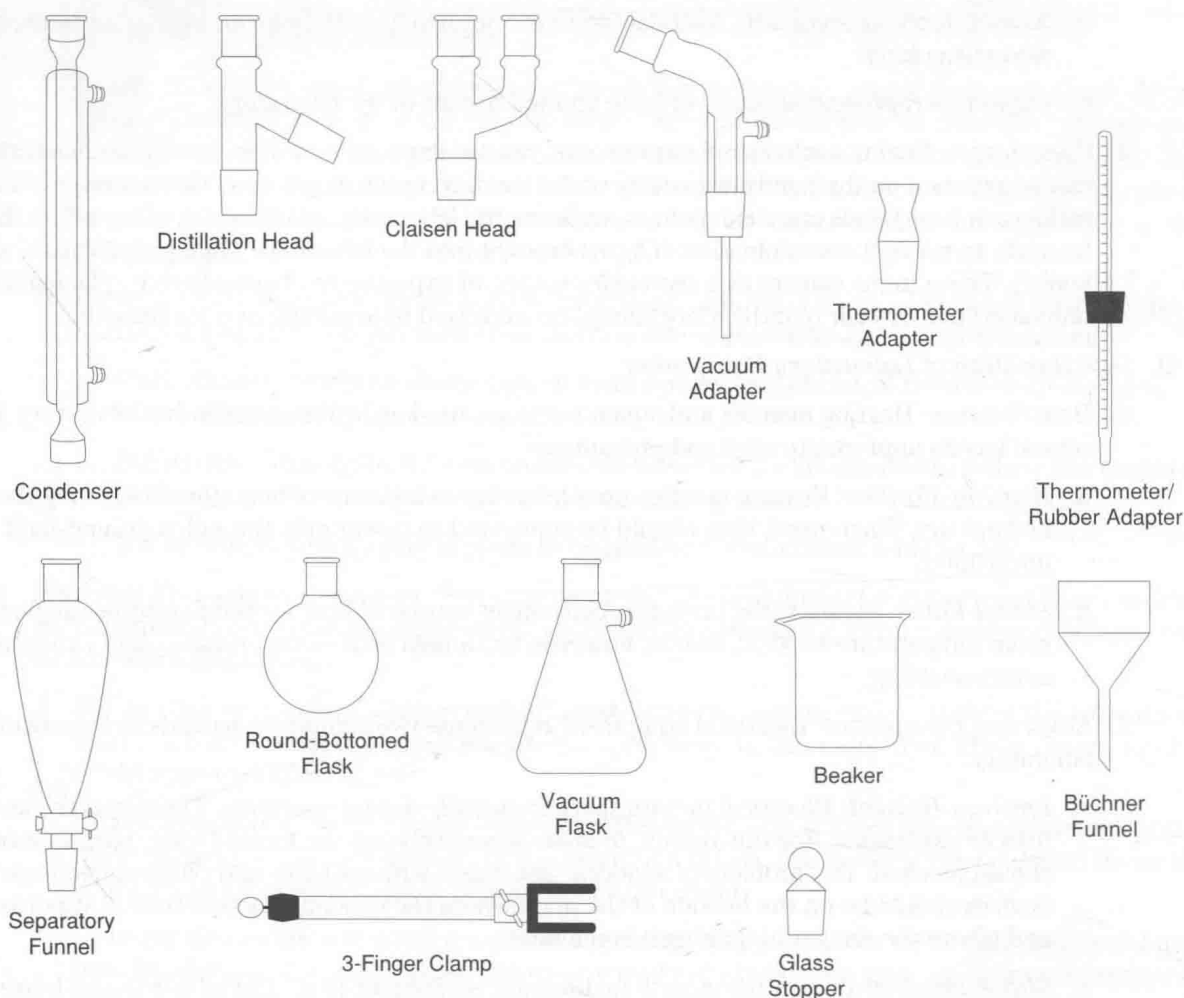


FIGURE 1.1
Chemical Glassware/Equipment with Joints

the experiment (see Figure 1.2). If a flaw is detected, the item should be sent to a glass blower for repair and not used in the experiment.

Prior to assembly, both portions of the ground glass joint should be greased lightly with stopcock grease. A small amount of grease should be applied to both portions of the joint and the joints rotated together to assure an even distribution of the lubricant. The joint should rotate smoothly and quietly, and the entire joint should be translucent. Avoid excessive grease; it will contaminate your products.

The apparatus should be assembled starting with the glassware closest to the laboratory bench, that is from the bottom up. The entire setup should be supported by clamps and rings, and all of the joints should fit smoothly together without scraping. The glass equipment is not designed to be self-supporting, and it may fall apart or break if undue stress is placed upon it.

- iii. *Cleaning:* Hot water, soap, and a test tube brush will remove most common contaminants from laboratory glassware. (NOTE: make sure that the contaminants have been quenched and will not react violently with these cleaning agents.) For more stubborn residues, acetone is a very effective cleaning agent. If the flask still refuses to come clean, do not try stronger reagents without consulting your instructor.

Occasionally, a joint will refuse to separate (i.e., freeze). If this occurs, do not attempt to separate it yourself; see your instructor.



FIGURE 1.2
Round-Bottomed Flask with a Star Crack

b. *Beakers and Erlenmeyer Flasks*

- i. *Storage:* Beakers and flasks should be stored in an upright, dry condition. Care should be taken to assure that they do not shift and bang against each other as a drawer is opened.
- ii. *Uses:* Both beakers and flasks are also used to carry out reactions. Generally, beakers are used when easy access to the reaction process is important. Flasks, with their narrower mouths, are used when it is important to restrict the loss of material by splashing or evaporation.
- iii. *Cleaning:* The same cleaning procedures should be followed for beakers and flasks that was suggested for ground glass equipment.

c. *Thermometers*

- i. *Storage:* Thermometers should be stored in their shipping case or placed in a drawer so that they cannot roll out when the drawer is opened. Loose thermometers should be stored flat with nothing placed on top of them.
- ii. *Uses:* Thermometers are used to measure the temperature of a system. The expected temperature should always fall within the range of the thermometer.

Prior to using a thermometer, it should be checked to see that the bead is not separated or that the stem is not cracked. If it is, replace it.

A thermometer is usually mounted in an apparatus by using a rubber adapter. Care should be exercised when inserting the thermometer into this adapter. It is very tempting to grasp the thermometer at the end farthest from the adapter and then push. Under these circumstances, the thermometer may snap and seriously gash the experimenter. It is safer to wrap the thermometer in a towel, grasp it close to the adapter, and then insert the thermometer in short segments. Under these circumstances, the thermometer seldom snaps. In the event the thermometer breaks, the instructor should be consulted for the proper cleanup procedure.

- iii. *Cleaning:* Thermometers should be removed from their adapters immediately after they have been used. The same precautions used to insert the thermometer should be used to remove it. They should also be cleaned with soap and water as recommended for other types of glassware.

d. *Graduated Cylinders*

- i. *Storage:* Graduated cylinders are generally stored upright in cabinets or on their sides in drawers.
- ii. *Uses:* Graduated cylinders are used to measure liquid volumes with reasonable precision.
- iii. *Cleaning:* Soap and water cleanup is usually adequate. Stubborn residues can be removed with acetone. Take care to assure that all water or solvent has been removed before reusing a graduated cylinder. A drying oven will remove any water that remains. Do not use a drying oven to remove acetone or other solvents; fires or explosions may result.

- e. *Disposable Glassware:* Pasteur pipettes and melting point capillaries are the most common types of disposable glassware.

- i. *Storage*: Both types of glassware are usually stored in bulk containers available in the laboratory. It is generally unnecessary to store these items in your equipment drawer.
- ii. *Uses*: Pasteur pipettes are used for the transfer of small amounts of liquids. Melting point capillaries are used to determine melting points.
- iii. *Cleaning*: Both items are usually discarded in a waste glass container. Generally, Pasteur pipettes are rinsed before being discarded.

f. *Funnels*

- i. *Storage*: Funnels are usually stored in a drawer, either on their sides or with the wide end down.
- ii. *Uses*: The two main types of funnels are: (1) gravity funnels and (2) suction funnels. Both types are used for separating solids and liquids.

Gravity funnels are generally V-shaped and are used to separate fairly coarse particulate matter from liquids. The driving force for the separation is gravity. The funnel, containing a folded piece of filter paper, is suspended over a beaker or flask. The solid-liquid mixture that needs to be separated is poured onto the filter paper, and the liquid is collected as it drips through.

Suction funnels usually contain a flat perforated plate. Filter paper, wet with solvent, is placed flat on the surface. This assembly is mounted on a vacuum flask. A vacuum is applied, and the solvent is drawn through the filter paper into the vacuum flask (see Figure 1.3). The driving force for this separation is the difference between atmospheric pressure and the vacuum in the flask.

Very fine solids can be separated from liquids using this technique. It is important to remember that the vacuum should be released by first carefully removing the rubber hose at the side arm of the vacuum flask; otherwise, the rush of air may spray the liquid.

- iii. *Cleaning*: Soap and water cleanups are usually sufficient to remove most residues. Stubborn residues should be removed with acetone. A drying oven can be used to remove any residual water, but never dry funnels that are wet with solvent or acetone in a drying oven; fires or explosions may result.

II. Laboratory Calculations/Record Keeping

Throughout this section, dimensional analysis will be applied to the calculations. In this process, certain numbers also have a dimension associated with them. In the first example, **density** has the dimensions of g/mL. In completing the calculations, these dimensions can be multiplied and divided just like numbers. The answer should have the correct dimensions. In the first example, the answer is grams. Incorrect dimensions indicate that some operation has not been conducted correctly.

A. *Interconversion of Masses and Volumes*

1. *Conversion of Volume to Mass for a Pure Liquid*: Throughout the text, amounts of pure liquid reagents are specified in volume measure (mL or L). To convert volume measure to masses (e.g., stoichiometry calculations), use the following formula:

$$\text{mass (g)} = \text{volume (mL)} \times \text{density (g/mL)}$$



FIGURE 1.3
Vacuum Filtration

Example: Assume that you have 20 mL of acetic anhydride ($d = 1.087 \text{ g/mL}$). How many grams of acetic anhydride are present?

Solution: $\text{mass (g)} = 20 \text{ mL} \times 1.087 \text{ g/mL} = 22 \text{ g}$

2. *Conversion of Volume to Mass for a Solute:* The calculation for the amount of solute in a solvent depends on the type of solution used. The concentration of the solutes is given in several different sets of units. These are weight/weight (w/w), weight/volume (w/v), and volume/volume (v/v).

- a. *Concentration Expressed as Weight/Weight:* A solution concentration may be expressed in terms of grams of solute per grams of solution. To calculate the amount of solute, use the following equation:

$$\text{Solute mass} = D \times V \times C$$

D = solution density in grams per mL

V = volume in mL

C = concentration in grams per 100 g of solution

Example: Calculate the amount of acetic acid present in 200 mL of a solution containing 7.5 g of acetic acid per 100 g of solution. The density of the solution is 1.3 g/mL.

Solution: $\text{Mass of acetic acid} = 200 \text{ mL} \times (7.5 \text{ g}/100 \text{ g}) \times 1.3 \text{ g/mL} = 20 \text{ g}$

- b. *Solution Concentration Expressed in Terms of Weight/Volume:* Weight/volume relationships are expressed in terms of molar concentrations or as mass of solute per unit volume of solution.

- i. *Concentrations Expressed in Terms of Molarity:* If the molar concentration of the solute is known, then the following equations are applicable:

$$\text{Solute mass} = M \times V \times MW$$

M = solute molarity in moles per liter or millimoles per milliliter

V = volume of solution in liters

MW = molecular weight of solute

Example: Calculate the amount of acetyl chloride present in 100 mL of a 1.5 M solution of acetyl chloride dissolved in hexane.

Solution: $\text{Mass of acetyl chloride} = 1.5 \text{ mole/L} \times .100 \text{ L} \times 79 \text{ g/mole} = 12 \text{ g}$

- ii. *Weight/Volume Solutions:* In these solutions, the concentration is expressed in terms of mass of solute/volume of solution. The following equation is used:

$$\text{Solute weight} = C \times V$$

C = concentration of solute in grams/unit volume of solution

V = volume of solution

Example: Calculate the amount of methyl alcohol present in 100 mL of a solution with a concentration of 8.5 g of methyl alcohol per 1000 mL of solution.

Solution: $\text{Mass of methyl alcohol} = (8.5 \text{ g of solute}/1000 \text{ mL of solution}) \times 100 \text{ mL of solution} = 0.85 \text{ g}$

- B. *Yield Calculations:* The quantity of desired material that can be obtained in a synthesis, based on stoichiometry, is called the **theoretical yield**. Organic reactions, however, do not always proceed to completion (as shown in a balanced equation). Competing reactions can consume one or more of the reactants, which reduces the amount of product obtained. In addition, many organic reactions involve equilibrium processes, and significant amounts of reactants may still be present at the "end" of a reaction. The amount of material obtained is called the **yield**. A measure of the efficiency of a particular reaction is called the **percentage yield**.

1. *Determination of the Theoretical Yield:* Several steps are necessary to calculate the theoretical yield. As an example, consider the conversion of ethyl alcohol to ethyl iodide using phosphorus triiodide.

The equation is as follows:



Assume that you are starting with 20 g of ethyl alcohol and 41.2 g of phosphorus triiodide.

a. Balance the above equation:



b. Calculate the number of grams of ethyl iodide produced using each reactant:

i. Using ethyl alcohol as the limiting reagent (i.e., the material present in smallest molar amount that controls the amount of product produced):

$$(20 \text{ g}/46 \text{ g/mole})(3/3)(156 \text{ g/mole}) = 68 \text{ g of ethyl iodide expected}$$

(g alcohol/mw alcohol) (moles product/moles alcohol) (mw product)

ii. Using phosphorus triiodide as the limiting reagent:

$$(41.2 \text{ g}/412 \text{ g/mole})(3/1)(156 \text{ g/mole}) = 46.8 \text{ g of ethyl iodide expected}$$

Equation 2 produces fewer grams of ethyl iodide; therefore, phosphorus triiodide is the limiting reagent, and 46.8 grams of ethyl iodide are expected to be produced in this reaction.

2. *Determination of the Actual Yield:* The actual yield is determined by the direct weighing of the product.

3. *Determination of the Percentage Yield:* The percentage yield is given by the following equation:

$$\% \text{ Yield} = (\text{Actual yield/Theoretical yield}) \times 100\%$$

Assume that in the previous reaction you produced 14 g.

$$\% \text{ yield} = (14 \text{ g}/46.8 \text{ g}) \times 100\% = 30\%$$

4. *Determination of Overall Percentage Yield:* In carrying out a synthetic scheme that utilizes more than one reaction, the efficiency of the whole sequence is expressed by the overall percentage yield.

To calculate the overall yield for a series of reactions, use the following equation:

$$(Y_1 \times Y_2 \dots Y_n) \times 100$$

Y_1 to Y_n ... are the percentage yields expressed as a decimal fraction for each step in the reaction.

Example: Assume that two reactions in a synthetic scheme proceed in 25% and 50% yield, respectively. Calculate the overall yield for the reaction series.



Solution: The overall yield is given by

$$(0.25 \times 0.50) \times 100 = 0.125 \times 100 = 12.5\% \text{ overall yield}$$

With reaction sequences encompassing many steps, the overall yield may be quite small, even though the yields of the individual steps are quite high (e.g., four consecutive 90% yields give an overall yield of 66%).

C. *The Laboratory Notebook:* Throughout this course you will perform experiments and record your observations in a notebook. This notebook will become a record of your laboratory work, your procedure, your observations, and the outcome of your experiments. These experiments are only valuable if they can be reproduced. To ensure the accurate recording of your results, a notebook must be maintained. The notebook's format can vary from institution to institution; however, certain basic features remain constant. The following is a list of those constant features:

1. The notebook must be sewn-bound. Loose-leaf ring binders and spiral notebooks are inappropriate, since pages can be easily removed or lost.