1.7 SAMPLE CALCULATIONS FOR NOTEBOOK RECORDS

Students frequently have difficulty in setting up Tables of Reactants and Products and calculating theoretical yields, so two hypothetical examples are provided for your reference.

Example 1

Problem

Consider the reaction shown in Equation 1.2. Assume that you are to use 5 g (7.8 mL) of 1-pentene and 25 mL of concentrated HBr solution. Prepare a Table of Reactants and Products, determine the limiting reagent, and calculate the theoretical yield for the reaction.

$$CH_2 = CHCH_2CH_2CH_3 + HBr \longrightarrow CH_3CH(Br)CH_2CH_2CH_3 \qquad (1.2)$$

Answer

First of all, note that the equation is balanced, because the "1" that signifies that 1 mole of each reactant will react to produce 1 mole of product is omitted by convention. Because an aqueous solution of HBr, rather than the pure acid, is being used, the amount of HBr present must be determined. Concentrated HBr is 47% by weight in the acid, and its density, d, is 1.49 g/mL, a value that would be recorded in the column headed "Other Data." Consequently, 25 mL of this solution contains 17.5 g of HBr (25 mL \times 1.49 g/mL \times 0.47). The needed data can then be entered into Table 1.1.

The limiting reagent is 1-pentene because theory requires that it and HBr react in a 1:1 molar ratio, yet they have been used in a ratio of 0.07:0.22. This means that no more than 0.07 mole of product can be formed, since theory dictates that the ratio between 2-bromopentane and 1-pentene also be 1:1. The calculation of the theoretical yield is then straightforward.

You may find it convenient to use units of milligrams (mg), microliters (μ L), and millimoles (mmol) instead of grams, milliliters, and moles, respectively, in performing measurements and calculations when small quantities of reagents are used, as is the case for microscale reactions. For example, let's consider how Table 1.1 would be modified if 0.1 g of 1-pentene and 0.5 mL of concentrated HBr solution were used. If the calculations were done in grams and moles, the entries

Table 1.1 Table of Reactants and Products for Preparation of 2-Bromopentane

Compound	M.M.	Volume Used (mL)	Weight Used (g)	Moles Used	Moles Required	Other Data	
1-Pentene	70.14	7.8	5	0.07	1	*	
HBr	80.91	25	17.5	0.22	1	*	
2-Bromopentane	151.05	†	+	+	1	*	

^{*}These entries have been intentionally omitted in this example.

[†]These entries are left blank because this line is for the product.

Limiting reagent: 1-pentene

Theoretical yield: $151.05 \text{ g/mol} \times 0.07 \text{ mol} = 10.5 \text{ g}$

Table 1.2 Table of Reactants and Products for Preparation of 2-Bromopentane

Compound	M.M.	Volume Used (mL)	Weight Used (g)	Mmols Used	Mmols Required	Other Data	
1-Pentene	70.14	0.16	100	1.4	1	*	
HBr	80.91	0.5	358	4.4	1	*	
2-Bromopentane	151.05	†	†	+	1	*	

^{*}These entries have been intentionally omitted in this example.

Limiting reagent: 1-pentene

Theoretical yield: $151.05 \text{ g/mol} \times 1.4 \text{ mol} \times 211 \text{ mg}$

under "Moles Used" would be 0.0014 and 0.4, respectively. Errors can arise when making such entries, because a zero may inadvertently be added or dropped.

This potential problem is less likely if you enter the data as milligrams and millimoles. If you recognize that 0.1 g of alkene is 100 mg and 0.5 mL of HBr solution contains 358 mg of HBr (0.5 mL \times 1490 mg/mL \times 0.48), the entries would be those shown in Table 1.2. You may then determine the limiting reagent and calculate the theoretical yield as in Example 1. Note that the necessary cancellation of units occurs when the molar mass is expressed in mg/mmol.

Although the volume of 1-pentene to be used is expressed in milliliters, you may be measuring out this amount with a device that is calibrated in microliters (μ L, 1 μ L = 10^{-3} mL). Thus, in the present example, you would be using 160 μ L of 1-pentene (0.16 mL \times 10^3 μ L/mL).

Example 2

Problem

Now consider the transformation illustrated in Equation 1.3. Assume that you are to use 7 mL of ethanol and 0.1 mL of concentrated $\rm H_2SO_4$ as the catalyst. Prepare a Table of Reactants and Products, determine the limiting reagent, and calculate the theoretical yield for the reaction.

$$2 CH3CH2OH - \frac{H2SO4}{(Catalytic amount)} \rightarrow CH3CH2OCH2CH3 + H2O (1.3)$$

Answer

As in the previous example, a volumetric measurement must first be converted to a weight. The density of ethanol is 0.789 g/mL, information that would be entered under the column headed "Other Data," so that means 5.5 g is being used. Table 1.3 can then be completed. Note that the catalyst, although recorded in the table, is not used in any of the calculations because, by definition, it is not consumed during the reaction. Including it should help remind the experimentalist that it is indeed required to make the reaction occur!

Calculation of the theoretical yield is performed as in Example 1, with the important exception that a factor of 0.5 is incorporated to adjust for the fact that only one-half mole of diethyl ether would be produced for each mole of ethanol that is used.

[†]These entries are left blank because this line is for the product.

Table of Reactants and Products for Preparation of Diethyl Ether

	M.M.	Volume Used (mL)	Weight Used (g)			Other Data
	46.07	7	5.5	0.12	2	*
scid acid	†	0.1	+	+	+	*
Died ether	74.12	‡	‡	‡	1	*

stries have been intentionally omitted in this example.

SAFE LABORATORY PRACTICE: OVERVIEW

There is little question that one of the most important abilities that you, the aspiring organic chemist, can bring to the laboratory is a sound knowledge of how to perform experimental work safely. But just knowing how to work safely is insufficient! You must also make a *serious* commitment to follow standard safety protocols. In other words, having the knowledge about safety is useless if you do not put that knowledge into practice. What you actually do in the laboratory will determine whether you and your labmates are working in a safe environment.

Chemistry laboratories are potentially dangerous because they commonly house flammable liquids, fragile glassware, toxic chemicals, and equipment that may be under vacuum or at pressures above atmospheric. They may also contain gas cylinders that are under high pressure. The gases themselves may or may not be hazardous-for example, nitrogen is not, whereas hydrogen certainly is-but the fact that their containers are under pressure makes them so. Imagine what happen if a cylinder of nitrogen fell and ruptured: You could have a veritable rocket on your hands, and, if the tank contained hydrogen, the "rocket" might even come equipped with a fiery tail! This is another way of saying all substances are hazardous under certain conditions.

for tunately the laboratory need be no more dangerous than a kitchen or bathbut this depends on you and your labmates practicing safety as you work. Should you observe others doing anything that is unsafe, let them know about π 🗷 friendly manner. Everyone will benefit from your action. We'll alert you recently to the possible dangers associated with the chemicals and apparatus will use so that you can become well trained in safe laboratory practice. Mastery of the proper procedures is just as important in the course as obtaining wields of pure products, and carefully reading our suggestions will assist you in achieving this goal. Some safety information will be contained in the text describing a particular experiment or in the experimental procedure itself. It will also appear in highlighted sections titled "Safety Alert." These are designed to draw your special attention to aspects of safety that are of particular importance. We urge you to read these sections carefully and follow the guidelines in them carefully. You will then be fully prepared to have the fun and fulfillment of the laboratory experience.

Trace entries are left blank for reactants that serve only as catalysts.

entries are left blank because this line is for the product.

[🛫] reagent: ethanol

 $rac{d}{dt}$ yield: 74.12 g/mol imes 0.12 mol \cdot 0.5 = 4.4 g

1.9 SAFETY: GENERAL DISCUSSION

We highlight here, in the form of a Safety Alert, some general aspects regarding safe practices in the laboratory. Such alerts will appear throughout the textbook and should be read carefully.

SAFETY ALERT



Personal Attire

- Do not wear shorts or sandals in the laboratory; the laboratory is not a beach!
 Proper clothing gives protection against chemicals that may be spilled accidentally. It is advisable to wear a laboratory coat, but in any case, the more skin that is protected by clothing the better.
- 2. Always wear safety glasses or goggles in the laboratory. This applies even when you are writing in your laboratory notebook or washing glassware, since nearby workers may have an accident. It is best not to wear contact lenses in the laboratory. Even if you are wearing eye protection, chemicals may get into your eyes, and you may not be able to get the contact lenses out before damage has occurred. Should you have to wear corrective glasses while working in the laboratory, make certain that the lenses are shatterproof. Wearing goggles over such glasses is recommended because the goggles give additional protection from chemicals entering your eyes from the sides of the lenses.
- 3. Wear suitable protective gloves, which may be made of latex or a plastic such as chloroprene, when working with particularly hazardous chemicals. Some reagents are especially hazardous if they come into contact with your skin. The ones you are most likely to encounter in the organic laboratory are concentrated acids and bases, and bromine and its solutions. Check with your instructor whenever you are uncertain whether you should be wearing gloves when handling reagents.

General Considerations

- Become familiar with the layout of the laboratory room. Locate the exits from the room and the fire extinguishers, fire blankets, eyewash fountains, safety showers, and first-aid kits in and near your workspace. Consult with your instructor regarding the operation and purpose of each of the safety-related devices.
- Find the nearest exits from your laboratory room to the outside of the building. Should evacuation of the building be necessary, use stairways rather than elevators to exit. Remain calm during the evacuation, and walk rather than run to the exit.
- 3. Become knowledgeable about basic first-aid procedures. The damage from accidents will be minimized if first aid is applied promptly. Read the section "First Aid in Case of an Accident" on the inside front cover of this book.
- 4. Never work alone in the laboratory. In the event of an accident, you may need the immediate help of a coworker. Should you have to work in the laboratory outside of the regularly scheduled periods, do so only with the express permission of your instructor and in the presence of at least one other person.
- 5. Before performing any experiment, you should perform a hazard analysis by reviewing the set-up for the experiment and the chemicals that will be used and identifying any potential hazards. You should consider how these hazards might be eliminated or mitigated and what you will do in case something goes wrong.

Apparatus and Chemicals

Always check carefully for imperfections in the glassware that you will be using. This should be done not only when checking into the laboratory for the first time but also when setting up the apparatus needed for each experiment. Look for cracks, chips, or other imperfections in the glass that weaken it. Use care in storing your glassware so that it is not damaged when you open or close the locker or drawer.

Pay particular attention to the condition of round-bottom flasks and condensers. The flasks often have "star" cracks (multiple cracks emanating from a central point) as a result of being banged against a hard surface. Heating or cooling a flask having this type of flaw may cause the flask to rupture with loss of its contents. This could result in a serious fire, not just loss of the desired product. To detect such cracks, hold the flask up to the light and look at all its surfaces closely. With respect to condensers, their most vulnerable points are the ring seals—the points where the inner tube and the water jacket of the condenser are joined. Special care must be given to examining these seals for defects, because if cracks are present water might leak into your apparatus and contaminate your product or, worse, cause violent reactions.

If you detect imperfections in your glassware, consult with your teacher immediately regarding replacement. Cracked or seriously chipped apparatus should always be replaced, but glassware with slight chips may still be safe to use.

- 2. Dispose of glassware properly. The laboratory should be equipped with a properly labeled special container for broken glassware and disposable glass items such as Pasteur pipets and melting-point capillaries. It is not appropriate to throw such items in the regular trash containers, because maintenance personnel may injure themselves while removing the trash. Broken thermometers are a special problem because they usually contain mercury, which is toxic and relatively volatile. There should be a separate, closed container for disposal of thermometers. If mercury has spilled as a result of the breakage, it should be cleaned up immediately. Consult with your instructor about appropriate procedures for doing so.
- Know the properties of the chemicals used in the experiments. Understanding these properties helps you to take the proper precautions when handling them and to minimize danger in case of an accident. Handle all chemicals with care.

Refer to MSDSs (Sec. 1.10) to learn about toxicity and other potential hazards associated with the chemicals you use. Most chemicals are at least slightly toxic, and many are very toxic and irritating if inhaled or allowed to come in contact with the skin. It is a good laboratory practice to wear suitable protective gloves when handling chemicals, and there may be times when it is imperative to do so. Your instructor will advise you if you do not need to use gloves.

Should chemicals come in contact with your skin, they can usually be removed by a thorough and *immediate* washing of the affected area with soap and water. Do *not* use organic solvents like ethanol or acetone to rinse chemicals from your skin, as these solvents may actually assist the absorption of the substances into your skin.

4. Avoid the use of flames as much as possible. Most organic substances are flammable, and some are highly volatile as well, which increases their potential for being ignited accidentally. Examples of these are diethyl ether, commonly used as a solvent in the organic laboratory, and acetone. Occasionally, open flames may be used for flame-drying an apparatus or distilling a high-boiling liquid. In such cases, a Safety Alert section will give special precautions for their use. Some general guidelines follow.

- a. Never use an open flame without the permission of your instructor.
- b. Never use a flame to heat a flammable liquid in an open container. Use a water or steam bath, hot plate, aluminum block, or similar electrical heat device instead. If a flammable liquid must be heated with an open flame, equip the container holding the liquid with a tightly fitting reflux condenser.

Information about the flammability of many commonly used organic solvents is provided in Table 3.1. Do not assume that a solvent is not flammable just because it is not listed in the table, however. In such cases, refer to the MSDSs (Sec. 1.10) or other sources to determine flammability.

- c. Do not pour flammable liquids when there are open flames within several feet. The act of transferring the liquid from one container to another will release vapors into the laboratory, and these could be ignited by a flame some distance away.
- d. Do not pour flammable water-insoluble organic solvents into drains or sinks. First of all, this is an environmentally unsound way to dispose of waste solvents, and second, the solvents may be carried to locations where there are open flames that could ignite them. Water-soluble solvents can be flushed down the drain if local regulations permit; consult with your instructor about this.
- 5. Avoid inhaling vapors of organic and inorganic compounds. Although most of the pleasant and unpleasant odors you encounter in everyday life are organic in nature, it is prudent not to expose yourself to such vapors in the laboratory. Work at a fume hood when handling particularly noxious chemicals, such as bromine or acetic anhydride, and, if possible, when performing reactions that produce toxic gases.
- 6. Never taste anything in the laboratory unless specifically instructed to do so. You should also never eat or drink in the laboratory, as your food may become contaminated by the chemicals that are being used.
- 7. Minimize the amounts of chemicals you use and dispose of chemicals properly. This aspect of laboratory practice is so important that we have devoted a portion of Section 1.10 to it. Read the relevant paragraphs carefully and consult with your instructor if there are any questions about the procedures.

1.10 SAFETY: MATERIAL SAFETY DATA SHEETS (MSDSs)

The variety and potential danger of chemicals used in the organic chemistry laboratory probably exceed that of any laboratory course you have had. It is imperative to understand the nature of the substances with which you are working. Fortunately, the increased emphasis on the proper handling of chemicals has led to a number of publications containing information about the chemical, physical, and toxicological properties of the majority of organic and inorganic compounds used in the experiments in this textbook. A comprehensive source is *The Sigma-Aldrich Library of Chemical Safety Data* (Reference 6), and it or similar compilations should be available in your library or some other central location. Alternatively, you can usually access MSDS information by typing the name of the chemical of interest into the

Name	Ether	Reviews and standards	OSHA standard-air: TWA 400 ppm.
Other names	Diethyl ether	Health hazards	May be harmful by inhalation, ingestion, or skin absorption. Vapor or mist is irritating to the eyes, mucous membranes, and upper respiratory tract. Causes skin irritation. Exposure can cause coughing chest pains, difficulty in breathing, and nausea, headache, and vomiting.
CAS Registry No.	60-29-7	First aid	In case of contact, immediately flush eyes or skin was copious amounts of water for at least 15 min while removing contaminated clothing and shoes. If inhaber remove to fresh air. If not breathing, give artificial respiration; if breathing is difficult, give oxygen. If ingested, wash out mouth with water. Call a physician.
Structure	(CH ₃ CH ₂) ₂ O	Incompatibilities	Oxidizing agents and heat.
МР	–116 °C	Extinguishing media	Carbon dioxide, dry chemical powder, alcohol, or polymer foam.
ВР	34.6 °C (760 torr)	Decomposition products	Toxic fumes of carbon monoxide, carbon dioxide.
FP	-40°C	Handling and storage	Wear appropriate respirator, chemical-resistant gloves, safety goggles, other protective clothing. Safety shower and eye bath. Do not breathe vapor. Avoid contact with eyes, skin, and clothing. Wash thoroughly after handling. Irritant. Keep tightly closed. Keep away from heat, sparks, and open flame. Forms explosive peroxides on prolonged storage. Refrigerate. Extremely flammable. Vapor may travel considerable distance to source of ignition. Container explosion may occur under fire conditions. Danger: Tends to form explosive peroxides, especially when anhydrous. Inhibited with 0.0001% BHT.
Appearance	Colorless liquid	Spillage	Shut off all sources of ignition. Cover with activated carbon adsorbent, place in closed containers, and take outdoors.
Irritation data	Human eye 100 ppm	Disposal	Store in clearly labeled containers until container is given to approved contractor for disposal in accordance with local regulations.
Toxicity data	Man, oral LDL ₀ 260 mg/kg		

Figure 1.3
Summary of MSDS for diethyl ether.

query box of your browser and using the associated search function. The data provided by these sources are basically summaries of the information contained in the MSDSs published by the supplier of the chemical of interest. Your instructor may be able to provide these sheets because by federal regulation an MSDS must be delivered to the buyer each time a chemical is purchased.

The information in an MSDS can be overwhelming. For example, the official MSDS for sodium bicarbonate is some six pages long. Even the summaries provided in most compilations are quite extensive, as illustrated in Figure 1.3, which

contains specific data for diethyl ether. Entries regarding the structure and physical properties of the compound, including melting point (mp), boiling point (bp), and flash point (fp), are included, along with its CAS (Chemical Abstracts Service) Registry Number, which is unique for each different chemical substance, and RTECS (Registry of Toxic Effects of Chemical Substances) number. Further data are provided concerning its toxicity, the permissible levels set by OSHA for exposure to it in the air you breathe (time-weighted average of 400 ppm), and possible health consequences resulting from contact with the compound. For diethyl ether, the entry for "Toxicity Data" represents the *lowest* recorded *lethal* concentration for ingestion of the chemical. Valuable information is also given regarding first-aid procedures, classes of substances with which diethyl ether reacts and thus is "incompatible" with, products of its decomposition, and materials suitable for extinguishing fires involving ether. Finally, protocols for safe handling and storage are included, along with procedures for disposing and cleaning up spills of diethyl ether.

Accessing MSDS information from commercial sources can be very time-consuming, although it is useful to refer to one or more of them if you need more complete MSDS information than is available online (see the online resources associated with this textbook) or if you are to use or produce a chemical that is not listed on it. We've developed the Web-based MSDSs to provide you with a rapid and convenient way to obtain important information on the chemicals you will be using or producing in the experimental procedures performed when using this textbook. The data we've provided online for this textbook are much more abbreviated than those in other sources, as seen in Figure 1.4. In developing our summaries of MSDS data, we've focused on just those data most relevant to your needs in the introductory organic laboratory.

We noted in the discussion of notebook formats (Sec. 1.6) that you may be required to summarize MSDS data in your laboratory book. This could be a daunting assignment, given the amount of information with which you might be faced, as illustrated in Figure 1.3. To assist you in doing this, we have provided one possible format for a summary in Figure 1.5. A summary for a particular chemical has to be provided only once and can be recorded at the end of your laboratory notebook on pages reserved for that purpose. Whenever the chemical is encountered in later experiments, you would only need to refer to the location of the summary of its MSDS information. *However*, you should reread the MSDS information so that you can continue to handle the chemical properly. This same recommendation applies if you have a file of MSDS-related printouts from the online resources for this textbook.

To summarize, you may think that reading about and recording data like those contained in Figures 1.3–1.5 is not a good investment of time. This is absolutely wrong! By knowing more about the chemicals that are used in the laboratory, you will be able to work safely and to deal with accidents, should they occur. The end result will be that you accomplish a greater amount of laboratory work and have a more valuable educational experience.

1.11 SAFETY: DISPOSAL OF CHEMICALS

The proper disposal of inorganic and organic chemicals is one of the biggest responsibilities that you have in the organic laboratory. Your actions, and those of your labmates, can minimize the environmental impact and even financial cost to

Diethyl Ether C ₄ H ₁₀ O											
CAS No.	PS	Color	Odor	FP	BP	MP	đ	17	٠D	5=	
60-29-7	Liquid	Colorless	Sweet	40	35	-116	0.7	6 Z	75 76 78	 e I	
	ypes of is/Exposures	Acute Hazards/Symptoms			Prevention				For Aid For		
Fire		Severe fire hazard, severe explosion hazard; may form explosive peroxides; vapors or gases may ignite at distant ignition sources.			No flames, no sparks, no contact with hot surfaces.			are associated from the character of the character provider.			
Inhalation Central nervous system depression with drowsiness, dizziness, nausea, headache, and lowering of the pulse and body temperature.			Ventilation, local exhaust.			Ressove from exposur- immediately and seek medical advice.					
Skin		Irritation, defatting, and drying of the skin.			Protective gloves and clothing.			Remove contaminated clothes/jewelry; thoroughly wash skin with soap and water; and seek medical advice			
Eyes Painful inflammation.			Safety goggles. Thoroughly flu with water for min, removing lenses if possil seek medical a immediately.			water for s removing s if possib medical ac	everal contact le, and				
Ingestion	depression Central nervous system depression with nausea, vomiting, drowsiness, dizziness; stomach may become promptly distended, which may hinder breathing.			Do <i>not</i> eat or drink in the laboratory.			Seek medical advice				
Carcinogenie	city	Not a know	n carcinogen.	<u> </u>	Mutag	enicity			्रेट ज्ञ्जाउट	.	

For more detailed information, consult the Material Safety Data Sheet for this compound.

Abbreviations: CAS No. = Chemical Abstracts Service Registry Number; PS = physical state; FP = flash point (°C); BP = po

Figure 1.4 Example of MSDS data provided online for this textbook.

your school for handling the waste chemicals that are necessarily produced in the experiments you do.

The experimental procedures in this textbook have been designed at a scale that should allow you to isolate an amount of product sufficient to see and manipulate, but they also involve the use of minimal quantities of reactants, solvents, and

Compound	Health Hazards, First Aid, Incompatibilities, Extinguishing Media, and Handling					
Diethyl Ether	May be harmful by inhalation, ingestion, or skin absorption. Avoid contact with eyes, skin, and clothing. In case of contact, immediately flush eyes or skin with copious amounts of water. Keep away from hot surfaces, sparks, and open flames. Extremely flammable. Vapor may travel considerable distance to source of ignition. If spilled, shut off all sources of ignition. Extinguish fire with carbon dioxide, dry chemical extinguisher, foam, or water.					

Figure 1.5
Abstract of MSDS for diethyl ether.

drying agents. Bear in mind, however, that minimizing the amounts of chemicals that are used is only the *first* part of an experimental design that results in the production of the least possible quantity of waste. The *second* part is to reduce the amounts of materials that you, the experimentalist, *define* as waste, thereby making the material subject to regulations for its disposal. From a legal standpoint, the laboratory worker is empowered to declare material as waste; that is, unneeded materials are not waste until you say they are! Consequently, a part of most of the experimental procedures in this textbook is reduction of the quantity of residual material that eventually must be consigned to waste. This means some additional time will be required for completion of the experiment, but the benefits—educational, environmental, and economic in nature—fully justify your efforts. The recommended procedures that should be followed are described under the heading Wrapping It Up.

How do you properly dispose of spent chemicals at the end of an experiment? In some cases this involves simply flushing chemicals down the drain with the aid of large volumes of water. As an example, solutions of sulfuric acid can be neutralized with a base such as sodium hydroxide, and the aqueous solution of sodium sulfate that results can safely be washed into the sanitary sewer system. However, the environmental regulations that apply in your particular community may require use of alternative procedures. Be certain to check with your instructor before flushing any chemicals down the drain!

For water-insoluble substances, and even for certain water-soluble constitution is not permissible under any circumstances, and other procedures soluble followed. The laboratory should be equipped with various containers for constitution of liquid and solid chemicals; the latter should not be thrown in a trash can be this exposes maintenance and cleaning personnel to potential danger, and is containers must be properly labeled as to where the put in them, because it is very important for safety and environmental reasons that different categories of spent chemicals be segregated from one another. The are likely to find the following types of containers in the organic laborated ardous solids, nonhazardous solids, halogenated organic liquids, hydromand oxygenated organic liquids. Each student must assume the responsations seeing that her or his spent chemicals go into the appropriate container; container; containers and of disposal be required.