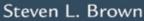


Laboratory Techniques for GENERAL CHEMISTRY











LABORATORY TECHNIQUES FOR GENERAL CHEMISTRY

Fourth Edition
Steven L. Brown



Copyright © 2012 by Steven L. Brown

Copyright © 2012 by Hayden-McNeil, LLC on illustrations provided

Photos provided by Hayden-McNeil, LLC are owned or used under license

Copyright © 2006 by Thomas Greenbowe and K.A. Burke for the SWH materials in Chapter 2 $\,$

All rights reserved.

Permission in writing must be obtained from the publisher before any part of this work may be reproduced or transmitted in any form or by any means, electronic or mechanical, including photocopying and recording, or by any information storage or retrieval system.

Printed in the United States of America

 $10\,9\,8\,7\,6\,5\,4\,3\,2\,1$

ISBN 978-0-7380-4472-9

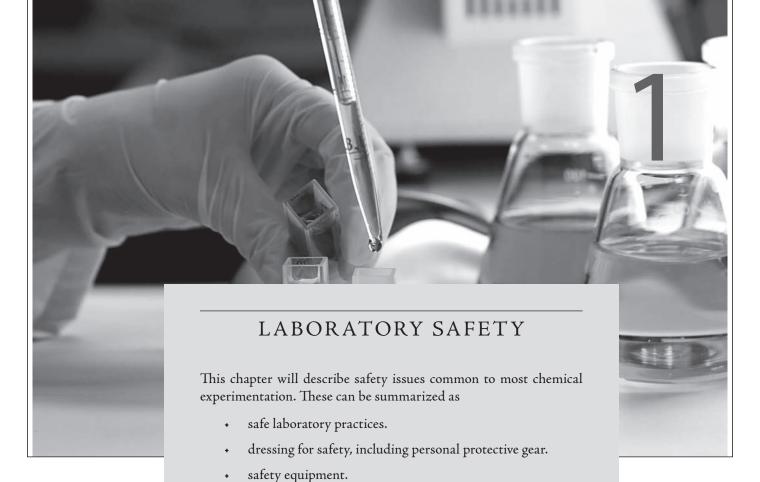
Hayden-McNeil Publishing 14903 Pilot Drive Plymouth, MI 48170 www.hmpublishing.com

Brown 4472-9 F11



TABLE OF CONTENTS

Chapter 1	Laboratory Safety
Chapter 2	Data Collection and Analysis
Chapter 3	Communicating Experimental Results
Chapter 4	Linear, Mass, and Volume Measurement
Chapter 5	Separation Techniques
Chapter 6	Chromatography
Chapter 7	Substance Characterization
Chapter 8	Spectroscopy
Chapter 9	Potentiometric Measurements
Chapter 10	Solution Properties
Chapter 11	Solution Preparation
Chapter 12	Chemical Synthesis
	Index



- handling chemicals.dealing with the unexpected.
- waste disposal.

By its nature the chemical laboratory can be a dangerous place to work. The improper use of chemicals and equipment can cause severe and/or permanent damage to your body. And you are not the only one at risk. Your actions can affect your coworkers, those who follow, and the environment. One of the more important aspects of your chemistry lab experience will be learning how to perform experiments as safely as is humanly possible. You will learn how to protect yourself and others from the hazardous conditions created by your experimentation.

This chapter covers general safety issues. Additional safety information will be presented with each experiment. Prior to each experiment your instructor will describe the proper use of chemicals and equipment. It is expected that you will become familiar with all this information. Your grade will depend, in part, on how well you demonstrate safe lab habits and knowledge of safe laboratory practices. You should expect to see the material presented here on the exams you take. In addition, your instructor's evaluation of your lab performance will be based, in part, on your adherence to safe laboratory practices and the lab rules. Finally, should you demonstrate a lack of regard for safety rules, or laboratory safety in general, you will be prohibited from working in the lab.

1-1 SAFE LABORATORY PRACTICES

It is common knowledge that doing chemistry is a hazardous activity. But so are cooking and driving a car. As with these other activities, the trick to a safe chemistry lab experience is to learn safe practices and develop safe working **habits**. This course is designed to teach safe lab practices and to help you develop habits that will lead to efficient, effective, and **safe** experimentation.

When performed correctly, the experiments presented in this course are safe. But this is a teaching lab. Mistakes will be made. Misinterpretation of the instructions or use of the wrong chemicals can result in a serious accident. And no matter how careful you are, the student working next to you may be doing something to endanger your health that you are helpless to prevent. No matter how much faith you have in your ability to work safely in the laboratory it is foolish to believe that an accident can't happen to you.

The safety rules are designed with this idea in mind. If you faithfully adhere to these rules, and to your instructor's precautions, you will find working in the chemical laboratory to be no more dangerous than working in a kitchen or driving a car.

Keep in mind that safety rules don't exist just to protect you and your lab mates. They are also intended to help instill safe working habits. Because habits are developed by repetition, it is expected that you will adhere to these rules at all times, regardless of the level of danger posed by any particular experiment.

An important component of a safe lab is cleanliness. A cluttered lab is a breeding ground for accidents. While performing an experiment, your work space should contain only those items that are currently needed. Spills should be immediately cleaned up. You must be aware at all times of the contents of all containers. At the end of the class you must turn off all water and gas jets, return all borrowed items and return all of your equipment to its proper storage location. You also must leave a clean workbench for the student that follows.

1-2 DRESSING FOR SAFETY



There are two dangers from chemical contact with skin:

- 1. destruction of tissue (chemical burns) and
- 2. poisoning due to absorption through the skin.

You should think about the materials you will wear to lab. Many fabrics, such as nylon and polyester, react with common lab chemicals. Most burn well. Cotton is the safest material to wear in a lab.

Clothing should be simple and loose fitting, but not too loose. You don't want to restrict your freedom of motion. Conversely, you don't want to accidentally dip a baggy sleeve into concentrated acid.

Lab Coat

To protect yourself against splashed chemicals and fumes, you should cover as much of your skin as is practical. A **lab coat** is ideal. Long pants are also highly recommended.

The selection of a lab coat should take into account the anticipated dangers of the lab. An appropriate lab coat will fit well—not too big or too small. The sleeves will be long enough to cover the arms, but not so long they get in the way. It will cover to below the knees.

Lab coats made of synthetic fabrics should NOT be used if there is a significant danger of fire. Synthetic fibers not only burn well but also melt and can stick to the skin. The best material for lab coats and all other clothing worn in a chemistry lab is cotton.

Once exposed to dangerous chemicals, a lab coat should be either properly cleaned or safely discarded.

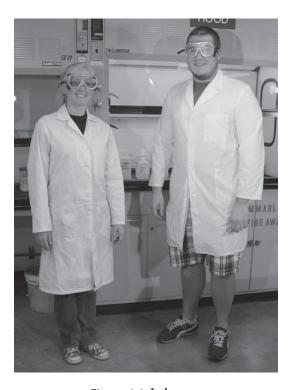


Figure 1-1. Lab coat.

Shoes

Because broken glass and spilled chemicals are common in the lab, nonabsorbent shoes that cover the entire foot are a must. Consider what would happen if you were wearing canvas sneakers and spilled acid on your shoe. It would soak through the shoe to your foot. In the time it would take you to get the shoe off, significant damage could be done. And it would be even worse if you were wearing socks.

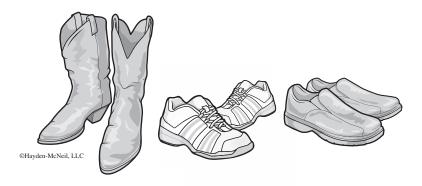


Figure 1-2. Acceptable footwear.

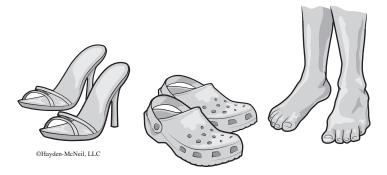


Figure 1-3. Unacceptable footwear.

Gloves



Hands present a special case. We use our hands to manipulate lab equipment and reagent bottles. This creates a large potential for inadvertent contact with chemicals, possibly resulting in chemical burns.

You will be instructed to wear gloves when you will be working with sufficiently dangerous chemicals. The procedures in this lab manual that require the use of gloves are indicated with the glove icon.

Not all gloves are suitable for use in a lab. The ones you select must not interfere with your ability to manipulate delicate glassware and instruments. They must also be impermeable to water and solvents. And they must be comfortable. The best choice for most lab situations are nitrile examination gloves (see Figure 1-4).



Figure 1-4. Lab gloves.

Eye Protection

Your eyes are quite possibly the most fragile parts of your body. They are certainly very important and impossible to replace. And they face many dangers in a chemical lab. *Proper protection of the eyes is mandatory in a chemical lab.*



Figure 1-5. Vented goggles.

While explosions pose the most dramatic danger to your eyes, they are not very common in chemistry labs. The greatest danger is from splashed chemicals. **Safety glasses** provide protection against particles propelled toward you (e.g., as the result of an explosion), but are not effective against splashes. Goggles with perforations are better, but still not adequate in many situations. Only **vented goggles** are considered adequate to properly protect you from splashed chemicals and flying particles.

1-3 LABORATORY SAFETY EQUIPMENT

Safety Showers

These provide first aid should you come into contact with large amounts of a dangerous chemical. You need to know the location and proper operation of the safety showers in your lab. To use the shower, stand under the head and pull the chain. Continue the washing for 15 minutes. Some showers are designed to dump a certain amount of water before they shut off.



Figure 1-6. A lab safety shower.

Eyewash Stations

These provide first aid in the event some foreign body or chemical enters the eye. You need to know the location and proper operation of the nearest eyewash station to your work space. Should you get a chemical in your eye, or should chemical vapors cause your eyes to water, immediately wash your eyes with lots of water. To be effective, eyewashing must be continuous for at least 15 minutes.



Figure 1-7. Eyewash fountain.

Fume Hoods

These are designed to prevent dangerous fumes, gasses, and vapors from entering the lab. They require a certain face velocity and are normally inspected periodically to insure they are functioning properly. For most chemical applications a minimal face velocity of 100 cubic feet per minute (cfm) is required. Modern hoods have flow rate indicators. If the air flow drops below the threshold value, an alarm sounds and an indicator light flashes.

Lab procedures that generate dangerous fumes should always be performed in a hood. Always close the sash as much as possible while still allowing comfortable working conditions.



Figure 1-8. Fume hood.

1-4 HANDLING CHEMICALS

All chemicals pose some level of hazard. Even something as common as water can be hazardous under the right conditions (consider someone who can't swim). It is critical that the hazards associated with all chemicals be known before the chemicals are used. The best source of information on chemical hazards is the **material safety data sheet**, commonly referred to as MSDS. A good place to find material safety data sheets is on the internet.

Chemicals, Solutions, and Reagents

In the chemistry lab these terms have specific meanings.

- Chemicals are single chemical compounds of known purity and composition.
 A chemical label will contain the name and purity. If no purity is given, it can be assumed to be 100%.
- Solutions consist of one chemical (the solute) dissolved in another (the solvent). A solution label will contain the name of the chemical, its concentration, and the solvent. If no solvent is named, it is assumed to be water.
- Reagents are all prepared chemicals and solutions used to detect, analyze, and make other substances. They can be as simple as a weak acid solution (e.g., 0.10 M HCl) or complex mixtures of a number of chemicals, such as Yamada's Universal Indicator, a solution of five different acid—base indicators.

Chemical Storage in the Lab

Low hazard chemicals, solutions, and reagents are stored on the **reagent bench**. This is also where you will find other supplies that you will need to perform your experiments.

Chemicals, solutions, and reagents that are expensive, rare, or require refrigeration are stored in the preproom. These must be checked out when needed.

More dangerous reagents are normally stored in a hood. This includes concentrated acids, concentrated bases, volatile liquids, and chemical waste.



Figure 1-9. A reagent bench.

Obtaining Reagents

Reagent bottles should not be removed from their storage location, especially those stored in a hood. The proper procedure for obtaining a reagent is to take an appropriate container to the reagent bottle and take only what you need. Transporting a large container of any chemical can be hazardous and is to be avoided.

Check the label carefully before you use a reagent. Use of the wrong reagent will ruin your experiment and could result in a serious accident. You also need to make sure you label everything you make and use in the lab. Many accidents result from assumptions made about unlabeled containers.

When using reagent bottles, you must be observant for spills. One common cause of chemical burns in a teaching lab is spilled reagent left on the **outside** of the bottle. It is easy to slop reagent down the outside of the bottle. If you do so, make sure you clean it up. And watch out for wet bottles!

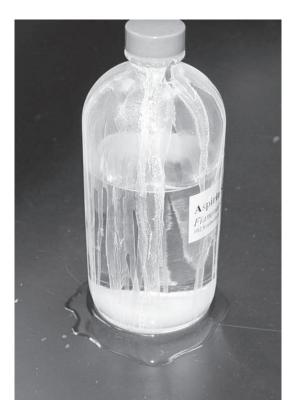


Figure 1-10. A dangerous bottle.

When using reagent bottles, you must also avoid contamination. You must develop habits that will minimize the possibility of contamination.

- NEVER set the cap to the reagent bottle down. Hold it between the second and third fingers of one hand.
- NEVER insert objects (including pipets) into reagent bottles. Instead, transfer some of the reagent to a clean beaker and pipet from this container. EXCEPTION: In those cases where there is a designated transfer pipet associated with the reagent bottle, this pipet may be inserted into the bottle.
- NEVER return unused reagent to the reagent bottle. Dispose of all unused reagent as chemical waste.



Figure 1-11. Holding a reagent cap while pouring reagent.

1-5 DEALING WITH THE UNEXPECTED

Even in the safest labs, the unexpected will occur. Breaking beakers and chemical spills are not uncommon. Sometimes these events lead to injuries. People get cut or burned. People pass out. Furthermore, no one is at their best when faced with the unexpected. Uncertainty or panic can make the situation worse. The best approach to any unexpected situation is to get help.

Should an accident occur, immediately notify the instructor.

Chemical contact can potentially lead to poisoning and/or tissue destruction. Poisoning occurs when chemicals are adsorbed through the skin. Chemical burns can be just as nasty as heat burns and often cause no pain until after damage is done. Any chemical contact with skin should be treated as a potential chemical burn. The first response to both is the same: Notify the instructor and run water over the exposed skin for 15 minutes.

Thermal burns are most commonly the result of contact with hot items such as a hot plate or hot beaker. Notify the instructor, run water over the exposed skin for 15 minutes and watch for blistering.

Minor cuts are usually the result of broken glass. Notify the instructor and wash with water until the bleeding stops. Even something as seemingly innocuous as a cut from a broken piece of glass could become very nasty if that glass were contaminated with a dangerous chemical. Let your instructor help judge the severity of any incident.

Major injuries and fainting are uncommon. Should such an event occur, get the instructor immediately.

Alarms sound when there is imminent danger. You will need to evacuate the building immediately. If you are performing an experiment, shut off all utilities (water, power, etc.) and then leave the lab.



Figure 1-12.

1-6 WASTE DISPOSAL

You will need to become familiar with the various kinds of waste generated in a chemistry lab and the proper protocol for disposing of each. These are:

Solid chemical waste. All solid chemicals remaining at the end of the experiment must be put in the designated container. Used transfer pipets are considered to be solid chemical waste and should be disposed of as such.

Liquid organic waste. A separate container is available for liquid organic waste. Never pour organics down the drain.

Acidic and basic water solutions. A separate container is provided for these. Never pour them down the drain.

Heavy metal water solutions. A separate container is provided for heavy metal waste. Never pour these solutions down the drain.

Glass waste. All glass waste must be put in the designated glass waste container. This includes glass with chemicals on it, such as capillary tubes, glass TLC plates, broken flasks, etc.

Garbage. Paper and other normal trash. Never put glass or chemicals in the garbage cans. Never put used transfer pipets in the garbage cans.

Drain water. Only neutral water solutions that do not contain heavy metals or organics can be poured down the drain. And never put any solids down the drain!



Figure 1-13. Chemical waste hood.

1-7 CHEMISTRY LAB SAFETY CHECKLIST

Before beginning work in the lab, you should make sure of the following:

- You have approved safety goggles and use them whenever any experimental work is being performed in the lab.
- · You have a lab coat and use it when required.
- · You are wearing an appropriate pair of shoes.
- You have gloves and use them when required.
- You are familiar with the precautions for the chemicals you will be using.
- · You know the location and proper operation of the following.
 - a. the hoods
 - b. the nearest safety shower
 - c. the nearest eyewash station
 - d. the first aid box
 - e. the exits
 - f. the reagent bench

1-8 STUDENT SAFETY AGREEMENT

You must agree to the following seven statements before you will be allowed to do any lab work in our labs. Please read them over and be prepared to indicate your agreement. If you have any concerns regarding these statements, you must discuss them with the course instructor before beginning lab work.

- I will obtain approved safety goggles and an appropriate lab coat and will wear them at all times when any experimental work is being done by anyone in the lab.
- I am familiar with the location and proper operation of the safety shower, the eyewash stations, and the first aid box.
- To the best of my ability, I will obey all instructions concerning the safe performance of experiments. I will use the hood when required, dispose of all chemicals and other materials as instructed, and promptly return all chemicals and reagents to their appropriate place when finished. I will not allow reagents or chemicals to become contaminated.
- I will protect myself by wearing appropriate clothing in the lab. I realize that
 I must wear shoes that protect my feet from chemical contact in the event of a
 spill.
- I will not attempt any unauthorized experiments nor will I work in the lab without proper supervision.
- I understand that my behavior in the lab is governed by the University's Code
 of Conduct and that failure to abide by University and Department safety
 rules and regulations will be considered a violation of that code and can result
 in my ejection from the lab.
- I understand that the University does not provide liability insurance coverage for me and that I am responsible for making arrangements to cover the financial burden of injuries I may incur during this class.