Sixth Edition

Laboratory Manual

General, Organic, and Biological Chemistry

Karen C. Timberlake

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ADDISON-WESLEY

This book is
dedicated to my husband, Bill,
and our son, John.
Thank you for your help, expertise, patience, and gourmet cooking
that make it possible for me to finish this book.

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Lanboratory Manual General, Organic, and Biological Chemistry, Sixth Edition

Some of the experiments in this lab book may be hazardous if materials are not handled properly or procedures are not followed correctly. Safety procedures of your college must be followed as directed by your instructor. Safety precautions must be utilized when you work with laboratory equipment, glassware, and chemicals.

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Preface

In this sixth edition of the Laboratory Manual, I have incorporated the helpful suggestions of my colleagues who have used the previous editions in their classes. It has been my desire to provide experiments that illustrate each of the chemical principles we discuss from the first day of class. I have also taken care to make each experiment workable as well as providing critical thinking for the student. The changes and goals of this laboratory manual address the following areas:

1. **Experiments are related to basic concepts of chemistry and health** Experiments are designed to illustrate the chemiocal principles that we discuss in our class. As often as possible experiments are related to health and medicine, and utilize common materials that are familiar to students.
2. **Experiments are flexible** Each experiment often includes 2-6 mini-experiments, which allows instructors to select those parts that accompany the weekly lecture and to fit the time frame that is available for lab (from 2 hours per week to 3 or 4 hours per week). Some of the experiments include extension activities to allow additional investigation in lab or sometimes outside of lab. There are now 42 labs with 26 lab labs on general chemistry, 8 on organic, and 8 on biochemistry. The biochemistry labs are longer and will probably require at least 2 lab sessions to complete. Some labs are paper labs (working safely, lab equipment, math review, and writing formulas) and can be used as outside of class activities.
3. **Safety** There is little or no use of hazardous materials. If a procedure involves some risk, a caution is included to remind students of safety procedures for laboratory work and to emphasize care in that step. Some experiments are recommend as instructor demonstrations.
4. **Experiment format provides clear instructions and evaluation** Students prepare for lab by reading goals, concepts to review, and pre-lab questions. Detailed background information discusses the chemistry concepts and give examples of calculations. Each experiment includes a large number of post-lab questions that relate to the concepts and request the students to do some calculations and to apply concepts to real life. The Lab Report pages allows for the names of groups of students in labs where students work collaboratively. Report pages ask students to obtain raw data, and incorporate it into graphing exercises, calculations, or conclusions about their results.
5. **Changes from previous edition** Experiments have been rewritten to run on a mini-scale level by reducing the amounts of chemicals needed. The safety information has been rewritten as experiment 1 and includes a safety quiz and safety commitment to be signed by the student. Experiment 2 now discuses laboratory equipment and the laboratory setup including a lab equipment quiz. The aim here is to highlight the safety and equipment preparation on the first day of lab (cannot usually do much else at the first lab). Some labs have been rewritten to fit the 2-3 hours time frame.
6. **Stock room preparation of chemicals** All the materials required for each experiment are listed in the appendices with amounts given for 20 students working in pairs. The experiments require standard laboratory equipment and chemicals. Most labs utilize common materials that are readily available and inexpensive. In some cases students bring samples from home.

I hope that this laboratory manual will help you in your chemistry instruction and that students will find they learn chemistry by participating in the laboratory experience.

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To The Student

Here you are in a chemistry laboratory with your laboratory book in front of you. Perhaps you have already been assigned a laboratory drawer, full of glassware and equipment you may have never seen before. Looking around the laboratory, you may see bottles of chemical compounds, balances, burners and other equipment that you are going to use. This may very well be your first experience with experimental procedures. At this point you may have some questions about what is expected of you. This laboratory manual is written with those considerations in mind.

The activities in this manual were written specifically to parallel the chemistry you are learning in the lecture portion of class. Many of the laboratory activities include materials that may be familiar to you, such as household products, diet drinks, cabbage juice, antacids and aspirin. In this way, chemical topics are related to the real world and to your own science experience. Some of the labs teach basic skills; others encourage you to extend your scientific curiosity beyond the lab.

It is important to realize that the value of the laboratory experience depends on the time and effort you invest in it. Only then will you find that the laboratory can be a valuable learning experience and an integral part of the chemistry class. The laboratory gives you an opportunity to go beyond the lectures and words in your textbook and experience the scientific process from which conclusions and theories concerning chemical behavior are drawn. In some experiments, the concepts are correlated with health and biological concepts. Chemistry is not an inanimate science, but one that helps us to understand the behavior of living systems.

Using this Laboratory Manual

Each experiment begins with learning goals to give you an overview of the topics you will be studying in that experiment. Each experiment is correlated to concepts you are currently hearing about in your chemistry class. Your instructor will indicate which activities you are doing. At the beginning of each experiment, you will also find a list of the materials needed for each activity.

The procedures are written to guide you through each laboratory activity. When you are ready to begin the lab, remove the report sheet at the end of the laboratory instructions. For each section on the report sheet, there is a set of procedures that tell you about the experimental activities. Read the procedures as you go. Measure carefully, report your date, follow instructions to complete the necessary calculations. You may also be asked to answer some or all of the follow up questions and problems designed to test your understanding of the concepts from the experiment.

It is my hope that the laboratory experience will help illuminate the concepts you are learning in the classroom. The experimental process can help make chemistry a real and exciting part of your life and provide you with skills necessary for your future.

Acknowledgments

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Table of Contents

EXPERIMENT 1

Working Safely in the Lab

Goals

Learn the safety rules for working with chemicals and participating in a safe manner when carrying out laboratory procedures.

Lab Questions

1. Why should students know the safety rules for doing experiments in a lab?
2. Why is it important for you to learn where the safety equipment is located in the lab?

Discussion

The chemistry laboratory with its equipment, glassware and chemicals has the potential for accidents. In order to avoid dangerous accidents, or to minimize their damage, precautions must be taken by every student to insure the safety of everyone working in the laboratory. Following the safety rules for handling chemicals and carrying our procedures will help to create a safe environment in the laboratory. Read the rules in the following sections and complete the questions in the laboratory report. Complete the safety quiz and sign and submit the commitment to lab safety.

Laboratory Activities

A. Preparing for Laboratory Work

1. Pre-read Before you begin an experiment, read the discussion and directions for that experiment. If you have been given a laboratory schedule, read the experiment ***before*** you come to the laboratory. Make sure you fully understand the experiment before starting the actual work. If you have a question, ask your instructor to clarify the procedures.

2. Do assigned work only Do only the experiments that have been assigned by your instructor. No unauthorized experiments are to be carried out in the laboratory. Experiments are done at assigned times, unless you have an open lab situation. Any change in procedure must be approved by your instructor. Do not work alone in a laboratory.

3. Wear proper clothing for protection For proper eye protection, safety goggles must be worn at all time in the lab. Wear sensible clothing to the laboratory. Loose sleeves, shorts, or open-toed shoes can be dangerous. A lab coat is useful in protecting clothes and covering arms. Wear shoes that cover the feet to prevent glass cuts and long pants and shirts to protect skin. Long hair should be tied back so it does not fall into chemicals or a flame from a Bunsen burner.

4. Safety awareness Learn the location and use of the emergency eye-wash fountains, the emergency shower, fire blanket, fire extinguishers, and exits. Memorize their locations in the laboratory. Be aware of other students in the lab carrying chemicals to their desk or to a balance. Do not use chipped or cracked glassware.

5. No food allowed *NO FOOD OR DRINK IS ALLOWED IN THE LABORATORY.* Wash your hands before you leave the laboratory. Do not let your friends visit while you are working in the lab; have them wait outside.

6. Prepare your work area Before you begin a lab, clear the lab bench or work area of all your personal items such as backpacks, books, sweaters, and coats. Find a storage place in the lab for them. All you will need is your laboratory manual, calculator, pen or pencil, text, and equipment from your lab drawer.

B. Handling Chemicals Safely

APPROVED EYE PROTECTION IS REQUIRED IN THE LABORATORY AT ALL TIMES

1. Eye injury Safety goggles must be worn all the time that you are in the laboratory. The particular type depends on state law, which usually requires industrial-quality eye protection. Contact lenses may be worn in the lab if needed for therapeutic reasons, provided that **safety goggles** are worn over the contact lenses. Contact lenses alone are dangerous because splashed chemicals make them difficult to remove. If chemicals accumulate under a lens, permanent eye damage can result. If a chemical should splash into your eyes, flood the eyes with water at the eye-wash fountain. Continue to rinse with water for at least ten minutes.

2. Check labels twice Be sure you take the correct chemical. ***DOUBLE CHECK THE LABEL*** on the bottle before you remove a chemical. For example, sodium sulfate could be mistaken for sodium sulfite if not read carefully. To avoid contamination of the chemical reagents, ***never*** insert droppers, pipets or spatulas into the reagent bottles.

3. Use small amounts Pour or transfer a chemical into a small, clean container (beaker, test tube, flask, etc.) taken from your lab drawer. Take only the quantity of chemical you need for the experiment and replace its cover. Do not keep a reagent bottle at your desk; ***return*** it to its proper location in the laboratory. Label the container. Many containers have etched sections on them for pencil. If not, use tape or a marking pencil.

4. Do not return chemicals to the original containers To avoid contamination of chemicals, dispose of used chemicals according to your instructor's instructions. ***Never return unused chemicals to reagent bottles.*** Some liquids and water-soluble compounds may be washed down the sink with plenty of water, but check with your instructor first. Dispose of organic compounds in specially marked containers in the hoods.

5. Do not taste chemicals; smell a chemical cautiously. Never use any equipment in the drawer such as a beaker to drink from. When required to note the odor of a chemical, first take a deep breath of fresh air and hold it while you use your hand to fan some vapors toward your nose and note the odor. Do not inhale the fumes directly. If a compound gives off an irritating vapor, use it in the fume hood to avoid exposure.

6. Do not shake laboratory thermometers Laboratory thermometers respond quickly to the temperature of their environment. Shaking a thermometer is unnecessary and can cause breakage.

7. Liquid spills Spills of water or liquids at your work area or floor should be cleaned up immediately. Small spills of liquid chemicals can be cleaned up with a paper towel. Large chemical spills must be treated with adsorbing material such as cat litter. Place the soaked material in a waste disposal bag and label. If a liquid chemical is spilled on the skin, flood *immediately with water* for at least 15 minutes. Any clothing soaked with a chemical must be removed since absorbed chemical can cause more damage.

8. Mercury spills The cleanup of mercury requires special attention. Notify your instructor immediately of any mercury spills so that special methods can be used to remove the mercury properly. Mercury spills may occur from broken thermometers. Place any free mercury and mercury clean-up material in special containers for mercury only.

9. Laboratory accidents Always notify your instructor of any chemical spill or accident in the laboratory. Broken glass can be swept up with a brush and pan and placed in a specially labeled container for broken glass. Cuts are the most common injuries in a lab. If a cut should occur, wash, elevate, and apply pressure if necessary. Inform your instructor for further medical attention.

10. Clean up Wash glassware as you work. Begin your cleanup 15 minutes before the end of the laboratory session. Return any borrowed equipment to the stockroom. Be sure that you always turn off the gas and water at your work area. Make sure you leave a clean desk. Check the balance you used. Clean up any spills in the area.

C. Heating Chemicals Safely

1. Heat only heat-resistant glassware Glassware marked Pyrex or Kimax can be heated. Other glassware will shatter if heated. To heat solids or liquids in a test tube, hold the tube in a holder at an angle - not upright - over the flame. Move the test tube continuously as you heat the sides and bottom. Never point the open end of the test tube at anyone, or look directly into it. Be careful when you pick up equipment you may just have heated. This might be an iron ring, a clay triangle, a test tube or beaker, or a crucible. A hot piece of iron or glass looks the same as it does as room temperature. Do not place hot objects on a balance. Let the object cool first.

2 . Flammable liquids Never heat a flammable liquid over an open flame. If heating is necessary, your instructor will indicate the use of a steam bath or a hot plate.

3. Never heat a stoppered container When a closed system is heated, it can explode as pressure inside builds.

4. Fire Small fires can be extinguished by covering them with a watch glass. If a larger fire is involved, use a fire extinguisher to douse the flames. *Do not direct a fire extinguisher at other persons in the laboratory.* Shut off gas burners in the laboratory. While working in a lab, long hair should be tied back away from the face. If clothing or hair catch on fire, get the student to the floor and roll into a fire blanket. A student may also be placed under the safety shower to extinguish flames. Cold water or ice can be applied to small burns. Do not use grease or other oil-based compound.

D. Waste Disposal

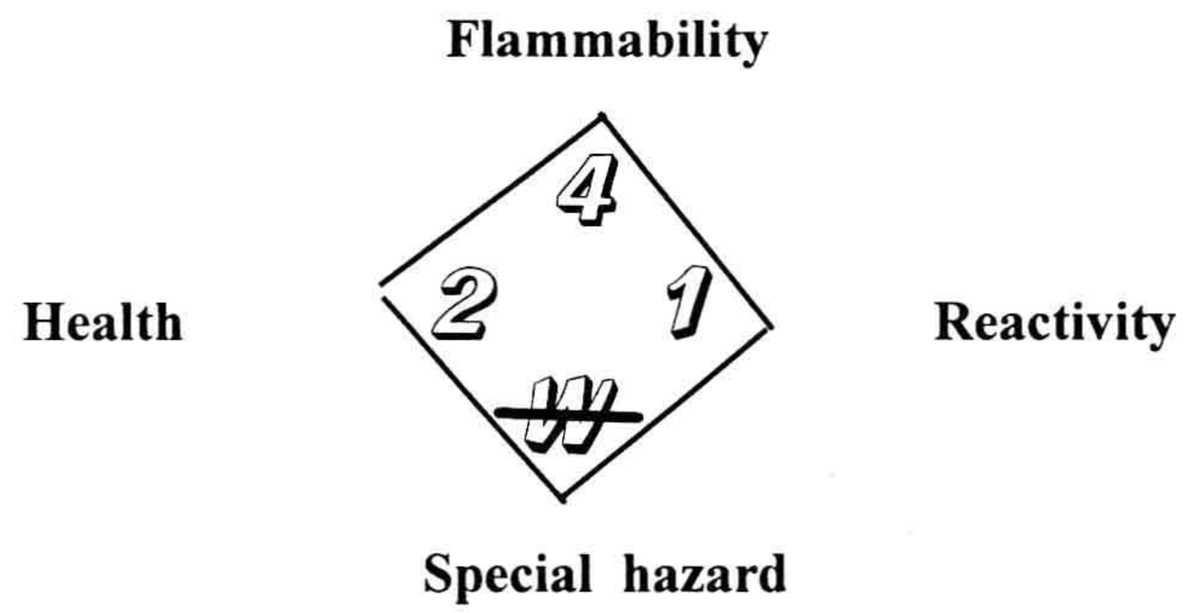
As you work in the laboratory, chemical wastes will be produced. Although we will use small quantities of materials, some waste products are unavoidable. In order to dispose of these chemical wastes safely, you will be informed of the specific disposal requirements by your instructor. Some of the general rules for chemical waste disposal follow.

1. Metals Metals should be placed in a container to be recycled.

2. Nonhazardous chemical wastes Substances such as sodium chloride (NaCl) that are soluble in water and are not hazardous may be emptied into the sink. If the waste is a solid, dissolve it in water before disposal.

3. Hazardous chemical wastes If a substance is hazardous or not soluble in water, it must be placed in a container that is labeled for waste disposal. Your instructor will inform you if chemical wastes are hazardous and identify the proper waste containers. ***If you are not sure about the proper disposal of a substance, ask your instructor.*** The labels on a waste container should indicate if the contents are hazardous, the name of the chemical waste, and the date that the container was placed in the lab.

4. **Hazard rating** The general hazards of a chemical are presented in a spatial arrangement of numbers with the flammability rating at twelve o'clock, the reactivity rating at the three o'clock position, and the health rating at the nine o'clock position. At the six o'clock position, information may be given on the reactivity of the substance with water. If there is unusual reactivity with water, the symbol W (do not mix with water) is shown. In the laboratory, you may see these ratings in color with blue for health hazard; red for flammability; and yellow for reactivity hazards.



A chemical is assigned a relative hazard rating based from one (little hazard) to four (extreme hazard). The health hazard indicates the likelihood of a material to cause injury due to exposure by contact, inhalation, or ingestion. The flammability hazard indicates the potential for burning. The reactivity hazard indicates the instability of the material by itself or with water with subsequent release of energy. Special hazards may be included such as W for reactivity with water or OX for oxidizing properties.

E. Safety Quiz and Pledge

Complete the safety quiz and check your answers with the key. Sign the commitment to lab safety and give to your instructor.

Name(s)_____ ***LABORATORY***

_____Section_____ ***REPORT 1***

Working Safely in the Lab

A. Preparing for Laboratory Work

Describe three things you can do to prepare for work in the laboratory.

1._____

2._____

3. _____

Diagram the location of the eye-wash fountain, the emergency shower, fire extinguisher(s), sinks, windows and exits.

B. Handling Chemicals Safely

Describe the safe way to carry out the following procedures in the lab.

1. Protecting your eyes

2. Bringing food into the lab

C. Heating Chemicals Safely

What precautions are taken for the following?

1. Glassware used for heating

2. Heating flammable liquids

D. Waste Disposal

1. How should you dispose of nonhazardous chemical wastes?

2. How should you dispose of hazardous chemical wastes?

E. Safety Quiz

Circle the correct answer(s) in each of the following questions. Some questions have more than one correct answer. Check your results in the answer key that is found at end of the safety quiz.

1. Approved eye protection is to be worn
   a. for certain experiments.
   b. only for hazardous experiments.
   c. all the time.

2. Eating in the laboratory is
   a. not permitted.
   b. allowed at lunch time.
   c. all right if you are careful.

3. If you need to smell a chemical, you should
   a. inhale deeply over the test tube.
   b. take a breath of air and fan the vapors toward you.
   c. put some of the chemical in your hand, and smell it.

4. When heating liquids in a test tube, you should
   a. move the tube back and forth through the flame.
   b. look directly into the open end of the test tube to see what is happening.
   c. direct the open end of the tube away from other students.

5. Unauthorized experiments are
   a. all right as long as they don't seem hazardous.
   b. all right as long as no one finds out.
   c. not allowed.

6. If a chemical is spilled on the you skin, you should
   a. wait to see if it stings.
   b. flood the area with lots of water for ten minutes.
   c. add another chemical to absorb it.

7. When taking liquids from a reagent bottle,
   a. insert a dropper.
   b. pour the reagent into a small container.
   c. put back what you don't use.

8. In the laboratory, open-toed shoes and shorts
   a. are OK if the weather is hot.
   b. are all right if you wear a lab apron.
   c. are dangerous and should not be worn.

9. When is it all right to taste a chemical?
   a. Never.
   b. When the chemical is not hazardous.
   c. When you use a clean beaker.

10. After you use a reagent bottle,
   a. keep it at your desk in case you need more.
   b. return it to its proper location.
   c. play a joke on your friends and hide it.

11. Before starting an experiment,
   a. read the entire procedure.
   b. ask your lab partner how to do the experiment.
   c. skip to the laboratory report and try to figure out what to do.

12. Working alone in the laboratory without supervision,
   a. is all right if the experiment is not too hazardous.
   b. is not allowed.
   c. is allowed if you are sure you can complete the experiment without help.

13. You should wash your hands
    a. only if they are dirty.
    b. before eating lunch in the lab.
    c. before you leave the lab.

14. Personal items (books, sweater, etc.)
    a. should be kept on your lab bench.
    b. should be left outside.
    c. should be stored out of the way, not on the lab bench.

15. When you have taken too much of a chemical, you should
    a. return the excess to the reagent bottle.
    b. store it in your lab locker for future use.
    c. discard it using proper disposal procedures.

16. In lab, you should wear
    a. sensible, protective clothing.
    b. something fashionable.
    c. shorts and loose sleeve shirts.

17. If a chemical is spilled on the table,
    a. clean it up right away.
    b. let the stockroom help clean it up.
    c. use appropriate absorbent if necessary.

18. If mercury is spilled,
    a. pick it up with a dropper.
    b. call your instructor.
    c. push it under the table where no one can see it.

19. If you hair or shirt catches on fire, you should
    a. run to the nearest exit.
    b. drop to the ground and roll.
    c. use the fire blanket to put it out.

20. A hazardous waste should be
    a. placed in a special waste container.
    b. washed down the drain.
    c. placed in the waste basket.

Answer Key to Safety Quiz

1. c	2. a	3. b	4. a, c
5. c	6. b	7. b	8. c
9. a	10. b	11. a	12. b
13. c	14. c	15. c	16. a
17. a, c	18. b	19. b, c	20. a