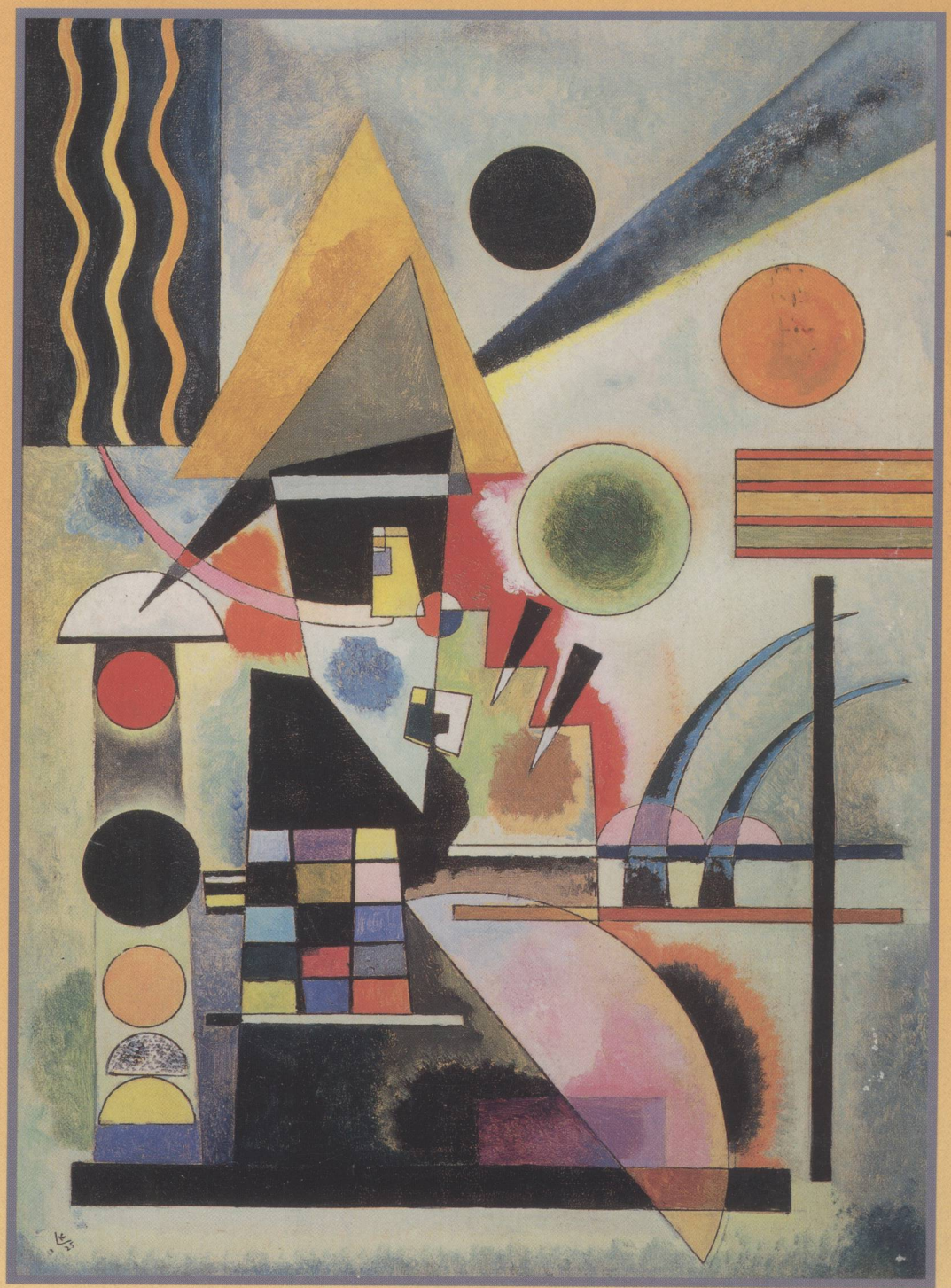
Laboratory Experiments for General, Organic, and Biochemistry

fourth edition



Bettelheim & Landesberg

Laboratory Experiments for General, Organic, and Biochemistry

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Warnings About Safety Precautions

Some of the experiments contained in this Laboratory Manual involve a degree of risk on the part of the instructor and student. Although performing the experiments is generally safe for the college laboratory, unanticipated and potentially dangerous reactions are possible for a number of reasons, such as improper measuring or handling of chemicals, improper use of laboratory equipment, failure to follow laboratory safety procedures, and other causes. Neither the publisher nor the authors can accept any responsibility for personal injury or property damage resulting from the use of this publication.

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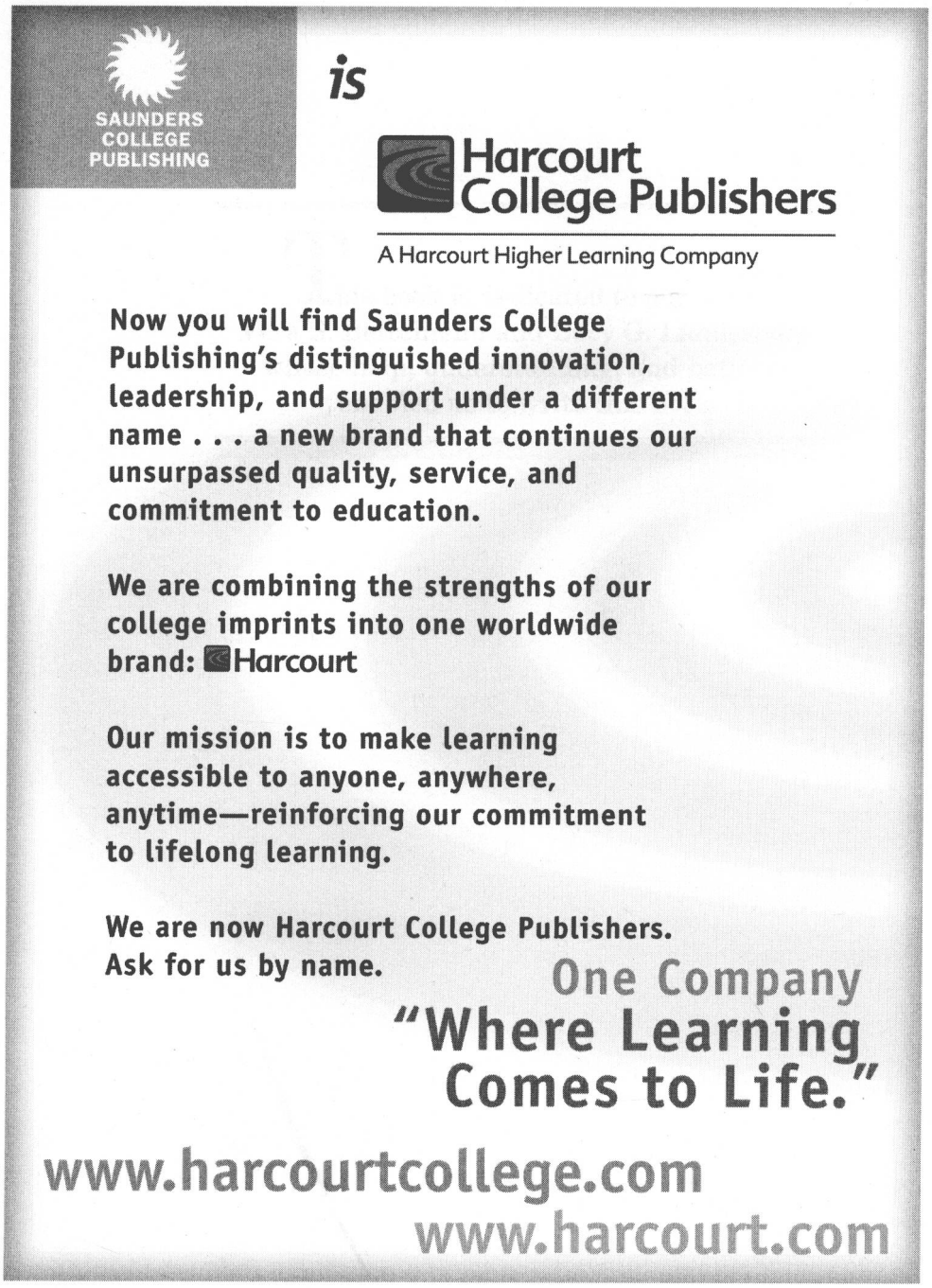
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This book is dedicated to our wives:
Vera S. Bettelheim and Lucy G. Landesberg,
whose help, understanding, and patience
enabled us to write this book.

Preface

In preparing the fourth edition of this Laboratory Manual, we wish to thank our colleagues who made this new edition possible by adopting our Manual for their courses. This fourth edition coincides with the publication of the sixth edition of the textbook: *Introduction to General, Organic and Biochemistry* by Bettelheim, Brown and March. The textbook shares the outline and the pedagogical philosophy with this book. As in previous editions, we have strived for the clearest possible writing in the procedures. The experiments give the student a meaningful, reliable laboratory experience that consistently work, while covering the basic principles of general, organic and biochemistry. Throughout the years, feedback from different Colleges and Universities made us aware that we have managed to achieve a manual that not only eases the student's task in performing experiments, but also is student friendly. Our new edition maintains this standard and improves upon it.

The major changes in this new edition are as follows: (1) We added two new experiments, thereby providing an even wider selection of experiments from which the instructor can choose. One of the new experiments demonstrates the Law of Constant Composition; the second provides for a water analysis. (2) We improved the procedures of all the experiments as a result of our observations of how our students carried out these experiments in our laboratories at Adelphi. (3) Safety issues and waste disposal are reemphasized throughout this edition. (4) We further improved on our aim to minimize the use of hazardous chemicals where possible and to design experiments that work on a semi-micro scale. (5) Most Pre-Lab and Post-Lab Questions have been changed or modified.

As in the previous editions, three basic goals were followed in all the experiments: (1) the experiments should illustrate the concepts learned in the classroom; (2) the experiments should be clearly and concisely written so that students will easily understand the task at hand, will work with minimal supervision because the manual provides enough information on experimental procedures, and will be able to perform the experiments in a $2^1/_2$-hr. laboratory period; (3) the experiments should not only be simple demonstrations, but also should contain a sense of discovery.

It did not escape our attention that in adopting this manual of Laboratory Experiments, the instructor must pay attention to budgetary constraints. All experiments in this manual require only inexpensive equipment, if any. A few spectrophotometers and pH meters are necessary in a number of experiments. A few experiments may require more specialized, albeit inexpensive, equipment, for example, a few viscometers.

The 52 experiments in this book will provide suitable choice for the instructor to select about 25 experiments for a two-semester or three-quarter course. The following are the principal features of this book:

1. The Table of Contents has been reorganized so that the first 25 experiments illustrate the principles of general chemistry, the next 12 those of organic chemistry, and remaining 15 those of biochemistry.
2. Each experiment starts out with background information that goes beyond the textbook material. All the relevant principles and their applications are reviewed in this background section.

3. The procedure part provides a step-by-step description of the experiments. Clarity of writing in this section is of utmost importance for successful execution of the experiments. **Caution!** signs alert the students when dealing with dangerous chemicals, such as strong acids or bases.

4. Pre-Lab Questions are provided to familiarize the students with the concepts and procedures before they start the experiments. By requiring the students to answer these questions and by grading their answers, we accomplish the task of preparing the students for the experiments.

5. In the Report Sheet we not only ask for the registration of the raw data, but we also request some calculations to yield secondary data.

6. The Post-Lab Questions are designed so that the student should be able to reflect upon the results, interpret them, and relate their significance.

7. At the end of the book in Appendix 3, we provide the Stockroom Personnel with detailed instructions on preparation of solutions and other chemicals for each experiment. We also give detailed instructions as to how much material is needed for a class of 25 students.

An Instructor's Manual that accompanies this book is **solely for the use of the Instructor.** It helps in the grading process by providing ranges of the experimental results we obtained from class use. In addition it alerts the instructor to some of the difficulties that may be encountered in certain experiments. The disposal of waste material is discussed for each experiment.

We hope that you will find our book of Laboratory Experiments helpful in instructing your students. We anticipate that students will like the book and find it inspiring in studying different aspects of chemistry.

Garden City, NY
April 2000

Frederick A. Bettelheim
Joseph M. Landesberg

Acknowledgments

These experiments have been used by our colleagues over the years and their criticism and expertise were instrumental in refinement of the experiments. We thank Stephen Goldberg, Robert Halliday, Cathy Ireland, Mahadevappa Kumbar, Jerry March, Sung Moon, Donald Opalecky, Reuben Rudman, Charles Shopsis, Kevin Terrance, and Stanley Windwer for their advice and helpful comments. We acknowledge the contributions of Dr. Jessie Lee, Community College of Philadelphia.

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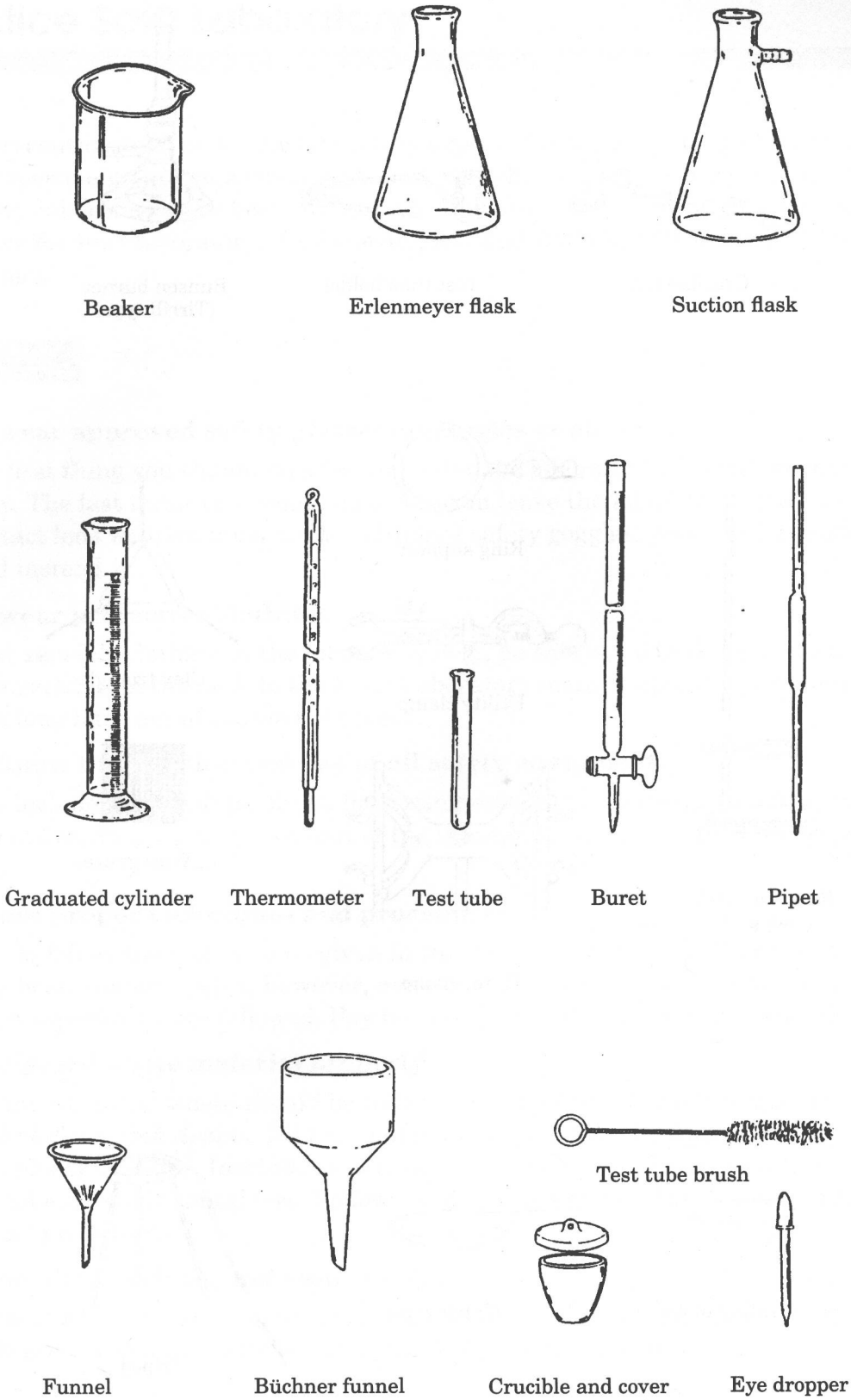


Figure 1 • Common laboratory equipment (From Weiner, S.A., and Peters, E.I.: *Introduction to Chemical Principles.* W.B. Saunders, Philadelphia, 1980.)

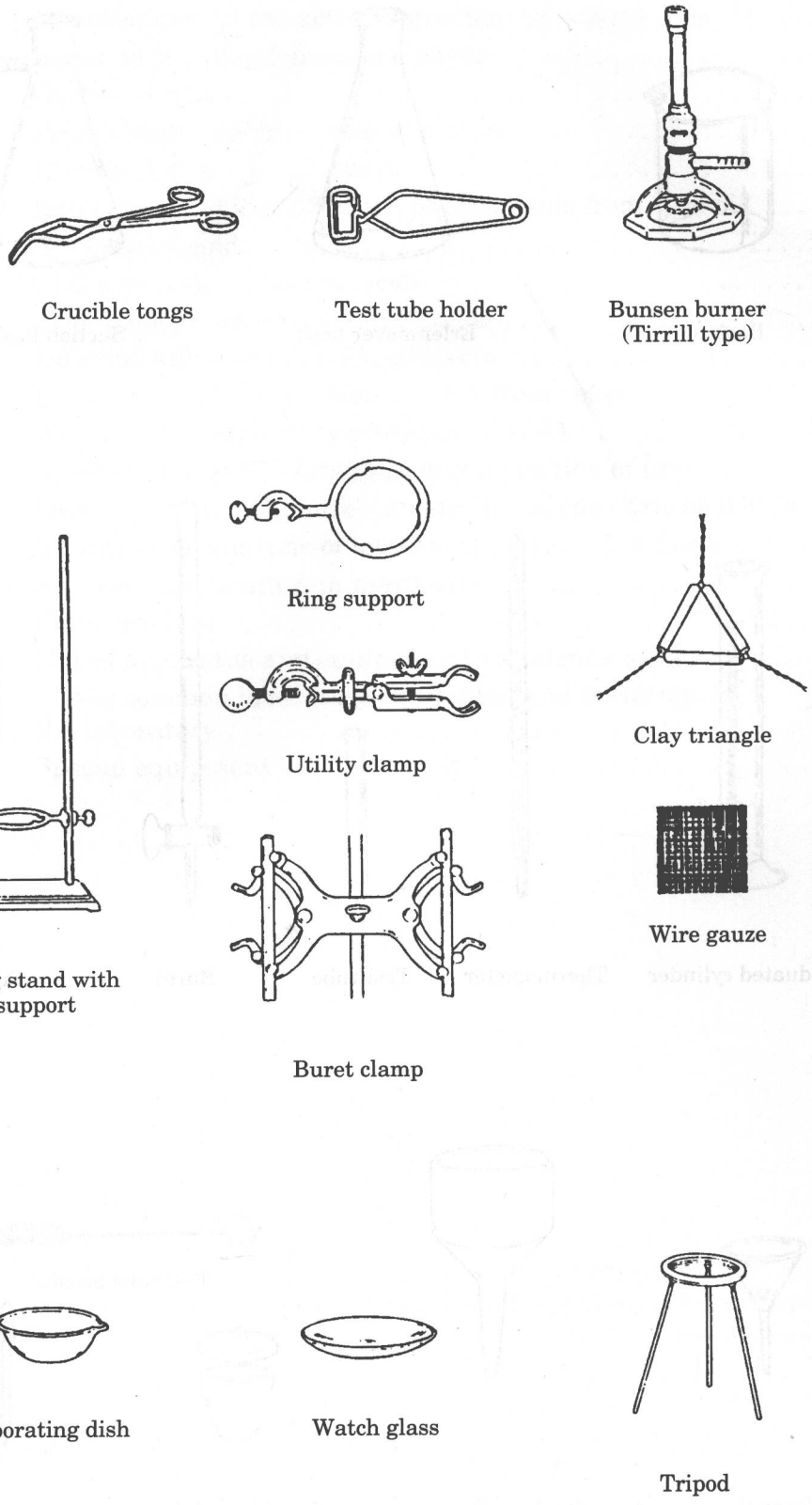


Figure 1 • (continued)

Practice Safe Laboratory

A few precautions can make the laboratory experience relatively hazard-free and safe. These experiments are on a small scale and as such, many of the dangers found in the chemistry laboratory have been minimized. In addition to specific regulations that you may have for your laboratory, the following **DO** and **DON'T RULES** should be observed at all times.

DO RULES

- ❑ **Do wear approved safety glasses or goggles at all times.**

  The first thing you should do after you enter the laboratory is to put on your safety eyewear. The last thing you should do before you leave the laboratory is to remove them. Contact lens wearers must wear additional safety goggles; prescription glasses can be used instead.

- ❑ **Do wear protective clothing.**

  Wear sensible clothing in the laboratory: e.g., no shorts, no tank tops, no sandals. Be covered from the neck to the feet. Laboratory coats or aprons are recommended. Tie back long hair, out of the way of flames.

- ❑ **Do know the location and use of all safety equipment.**

  This includes eyewash facilities, fire extinguishers, fire showers, and fire blankets. In case of fire, do not panic, clear out of the immediate area, and call your instructor for help.

- ❑ **Do use proper techniques and procedures.**

  Closely follow the instructions given in this laboratory manual. These experiments have been student tested; however, accidents do occur but can be avoided if the steps for an experiment are followed. Pay heed to the **Caution!** signs in a procedure.

- ❑ **Do discard waste material properly.**

  Organic chemical waste should be collected in appropriate waste containers and *not flushed down sink drains*. Dilute, nontoxic solutions may be washed down the sink with plenty of water. Insoluble and toxic waste chemicals should be collected in properly labeled waste containers. Follow the directions of your instructor for alternative or special procedures.

- ❑ **Do be alert, serious, and responsible.**

  The best way you can prepare for an experiment is to read the procedure carefully and be aware of the hazards before stepping foot into the laboratory.

DON'T RULES

- ❑ **Do not eat or drink in the laboratory.**

  Consume any food or drink before entering the laboratory. Chemicals could get into food or drinks, causing illness. If you must take a break, wash your hands thoroughly before leaving.

- ❑ **Do not smoke in the laboratory.**

  Smoke only in designated smoking areas outside the laboratory. Flammable gases and volatile flammable reagents could easily explode.

- ❑ **Do not taste any chemicals or breathe any vapors given off by a reaction.**

  If there is a need to smell a chemical, you will be shown how to do it safely.

- ❑ **Do not get any chemicals on your skin.**

  Wash off the exposed area with plenty of water should this happen. Notify your instructor at once. Wear gloves as indicated by your instructor.

- ❑ **Do not clutter your work area.**

  Your laboratory manual and the necessary chemicals, glassware, and hardware are all that should be on your benchtop. This will avoid spilling chemicals and breaking glassware.

- ❑ **Do not enter the chemical storage area or remove chemicals from the supply area.**

  Everyone must have access to the chemicals for the day's experiment. Removal of a chemical from the storage or supply area only complicates the proper execution of the experiment for the other students.

- ❑ **Do not perform unauthorized experiments.**

  Any experiment not authorized presents a hazard to any person in the immediate area.

- ❑ **Do not take unnecessary risks.**

These **DO** and **DON'T RULES** for a safe laboratory are not an exhaustive list, but are a minimum list of precautions that will make the laboratory a safe and fun activity. Should you have any questions about a hazard, ask your instructor *first*—not your laboratory partner. Finally, if you wish to know about the dangers of any chemical you work with, read the Material Safety Data Sheet (MSDS). These sheets should be on file in the chemistry department office.

Table of Contents

Experiment 1

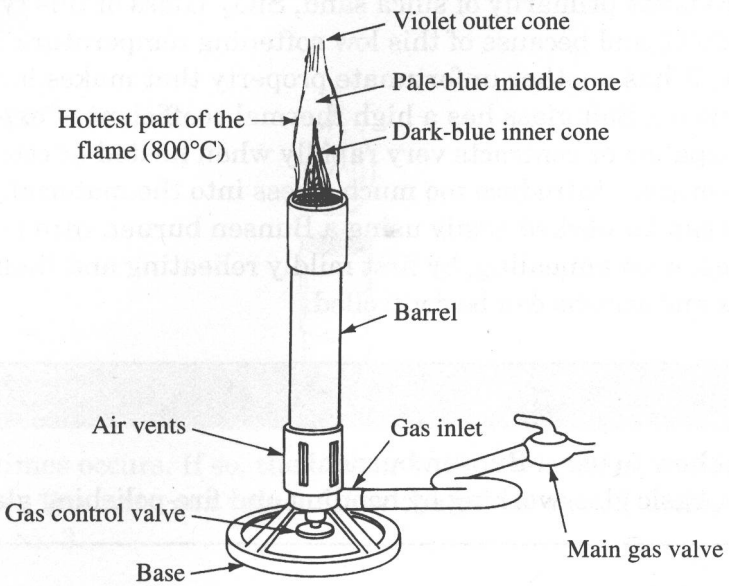
Laboratory techniques: use of the laboratory gas burner; basic glassworking

Background

The Laboratory Gas Burner

Tirrill or Bunsen burners provide a ready source of heat in the chemistry laboratory. In general, since chemical reactions proceed faster at elevated temperatures, the use of heat enables the experimenter to accomplish many experiments more quickly than would be possible at room temperature. The burner illustrated in Fig. 1.1 is typical of the burners used in most general chemistry laboratories.

Figure 1.1
The Bunsen burner.



A burner is designed to allow gas and air to mix in a controlled manner. The gas often used is "natural gas," mostly the highly flammable and odorless hydrocarbon methane, CH_4. When ignited, the flame's temperature can be adjusted by altering the various proportions of gas and air. The gas flow can be controlled either at the main gas valve or at the gas control valve at the base of the burner. Manipulation of the air vents at the bottom of the barrel allows air to enter and mix with the gas. The hottest flame has a violet outer cone, a pale-blue middle cone, and a dark-blue inner cone; the air vents, in this case, are opened sufficiently to assure complete combustion of the gas. Lack of air produces a cooler, luminous yellow flame. This flame lacks the inner cone and most likely is smoky, and often deposits soot on objects it contacts. Too much air blows out the flame.

Basic Glassworking

In the chemistry laboratory, it is often necessary to modify apparatus made from glass or to connect pieces of equipment with glass tubing. Following correct procedures for working with glass, especially glass tubing, is important.

Glass is a super-cooled liquid. Unlike crystalline solids which have sharp melting points, glass softens when heated, flows, and thus can be worked. Bending, molding, and blowing are standard operations in glassworking.

Not all glass is the same; there are different grades and compositions. Most laboratory glassware is made from borosilicate glass (containing silica and borax compounds). Commercially, this type of glass is known as *Pyrex* (made by Corning Glass) or *Kimax* (made by Kimble glass). This glass does not soften very much below 800°C and, therefore, requires a very hot flame in order to work it. A Bunsen burner flame provides a hot enough temperature for general glassworking. In addition, borosilicate glass has a low thermal coefficient of expansion. This refers to the material's change in volume per degree change in temperature. Borosilicate glass expands or contracts slowly when heated or cooled. Thus, glassware composed of this material can withstand rapid changes in temperature and can resist cracking.

Soft glass consists primarily of silica sand, SiO_2. Glass of this type softens in the region of 300–400°C, and because of this low softening temperature is not suitable for most laboratory work. It has another unfortunate property that makes it a poor material for laboratory glassware. Soft glass has a high thermal coefficient of expansion. This means that soft glass expands or contracts very rapidly when heated or cooled; sudden, rapid changes in temperature introduce too much stress into the material, and the glass cracks. While soft glass can be worked easily using a Bunsen burner, care must be taken to prevent breakage; with annealing, by first mildly reheating and then uniformly, gradually cooling, stresses and strains can be controlled.

Objectives

1. To learn how to use a Bunsen burner.
2. To learn basic glassworking by bending and fire-polishing glass tubing.

Procedure

The Laboratory Gas Burner; Use of the Bunsen Burner

1. Before connecting the Bunsen burner to the gas source, examine the burner and compare it to Fig. 1.1. Be sure to locate the gas control valve and the air vents and see how they work.
2. Connect the gas inlet of your burner to the main gas valve by means of a short piece of thin-walled rubber tubing. Be sure the tubing is long enough to provide some slack for movement on the bench top. Close the gas control valve. If your burner has a screw-needle valve, turn the knob clockwise. Close the air vents. This can be done by rotating the barrel of the burner (or sliding the ring over the air vents if your burner is built this way).

3. Turn the main gas valve to the open position. Slowly open the gas control valve counterclockwise until you hear the hiss of gas. Quickly strike a match or use a gas striker to light the burner. With a lighted match, hold the flame to the top of the barrel. The gas should light. How would you describe the color of the flame? Hold a Pyrex test tube in this flame. What do you observe?

4. Carefully turn the gas control valve, first clockwise and then counterclockwise. What happens to the flame size? (If the flame should go out, or if the flame did not light initially, shut off the main gas valve and start over, as described above.)

5. With the flame on, adjust the air vents by rotating the barrel (or sliding the ring). What happens to the flame as the air vents open? Adjust the gas control valve and the air vents until you obtain a flame about 3 or 4 in. high, with an inner cone of blue (Fig. 1.1). The tip of the pale blue inner cone is the hottest part of the flame.

6. Too much air will blow out the flame. Should this occur, close the main gas valve immediately. Relight following the procedure in step 3.

7. Too much gas pressure will cause the flame to rise away from the burner and "roar" (Fig. 1.2). If this happens, reduce the gas flow by closing the gas control valve until a proper flame results.

Figure 1.2
The flame rises away from the burner.

8. "Flashback" sometimes occurs. If so, the burner will have a flame at the bottom of the barrel. Quickly close the main gas valve. Allow the barrel to cool. Relight following the procedures in step no. 3.

Basic Glassworking; Working with Glass Tubing

Cutting glass tubing

1. Obtain a length of glass tubing (5–6 mm in diameter). Place the tubing flat on the bench top, and with a grease pencil mark off a length of 30 cm. Grasp a triangular file with one hand, placing your index finger on a flat side of the file. With your other hand, hold the tubing firmly in place against the bench top. At the mark, press the edge of the file down firmly on the glass, and in one continuous motion scratch the glass (Fig. 1.3).

Figure 1.3
Cutting glass tubing with a triangular file.