3RD EDITION

FRANTZ/MALM'S ESSENTIALS OF CHEMISTRY IN THE LABORATORY

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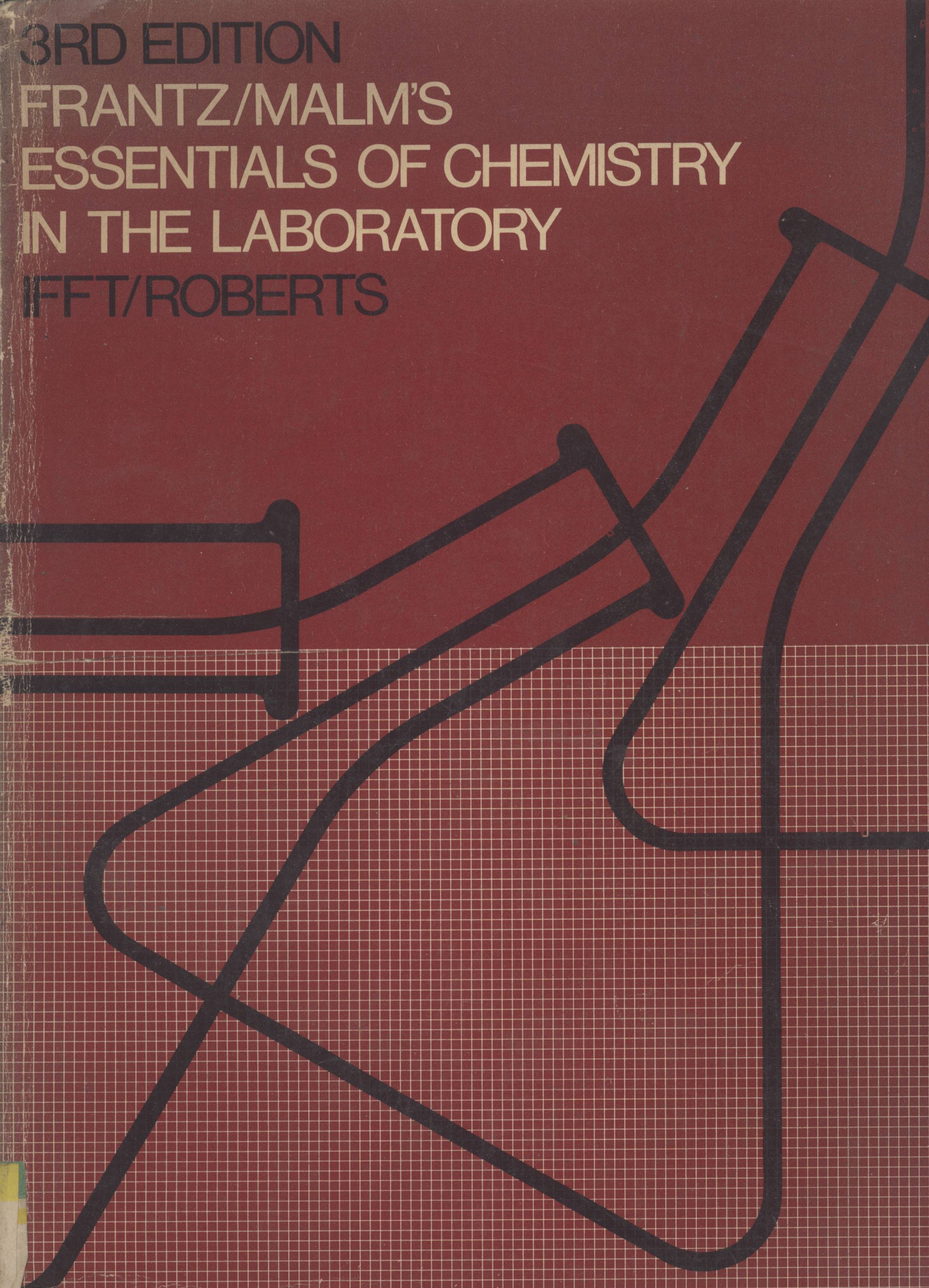

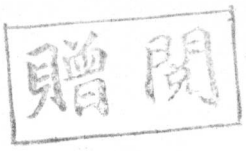

THIRD EDITION

FRANTZ/MALM'S
Essentials of Chemistry in the Laboratory

James B. Ifft
Julian L. Roberts, Jr.

UNIVERSITY OF REDLANDS

W. H. FREEMAN AND COMPANY
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Freeman Laboratory Separates in General Chemistry

Each exercise in this manual is available as a Freeman Laboratory Separate, numbered below in the order in which they appear in the manual.

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To
Fern, Frances, Evelyn, and Jane

PREFACE

Today's society is confronting unprecedented challenges. Almost every day newspapers feature major items on the problems we face—energy shortages, overpopulation, pollution of our air and water, food shortages, and so on. To be sure, scientists themselves have contributed to some of these problems, but they—along with politicians, economists, and sociologists—will be called upon to help resolve all of them.

Chemistry is one of those natural sciences being applied to the quest for a better environment and better living standards for all people. Whether chemists are devising new and safer pesticides, more effective birth control measures, techniques to monitor and reduce air pollution, or a cure for cancer, their expertise is urgently needed by society.

We assume that the student is taking a laboratory course in chemistry, first because he thinks an acquaintance with this particular science can contribute to his understanding of the physical world and, second, because the skills he acquires can help him to earn a living. But we hope that he is taking it also because he realizes that a knowledge of this subject will enable him to make a positive contribution to the solution of some of the grave problems currently facing all of us.

Purpose. Some general chemistry instructors are dropping the laboratory portion of their courses. Most of these decisions are pragmatic, resulting from the very real difficulty of providing a laboratory experience for large numbers of students. It costs both money and time. We believe that it is worth every dollar and every hour.

In this new edition for the Frantz/Malm series of laboratory manuals, the basic premise continues to be that the laboratory part of the course must be a central aspect of the student's learning experience. The relationship between the lecture and laboratory portions of the course should therefore be closely coordinated and mutually supportive.

One reason is that chemistry *is* an experimental science. Although some theoretical calculations can be performed with complete rigor, vast areas of

kinetics, quantum mechanics, and descriptive chemistry rest principally on the work of men and women conducting experiments at the benchtop.

Also, laboratory work is fun. The appearance of a precipitate at the right stage in a qualitative analysis experiment, the striking rainbow of colors from a series of indicators at a series of *p*H's, or the resolution of the light from a lamp into a series of lines with the use of a spectroscope one has made himself—all are stimulating and rewarding experiences in themselves.

Finally, we believe that conducting experiments in the chemistry laboratory is the best way to learn descriptive chemistry. Few students will forget the surprise with which they watch their results the first time they add base to a sodium dichromate solution, or add silver nitrate to a solution containing chloride ion, or reach the end point in the titration of an acid with a base using phenolphthalein as the indicator. One's own visual experiences, then, are definite aids to learning and retaining the inorganic and organic material described in the standard chemistry text.

Organization and Content. The organization of this manual has been revised somewhat in order to reflect changing course content and student needs. First, the arrangement of the experiments is different. For instance, all of the experiments on gases have been combined into one section, and all acid-base experiments are contained in a single section ("Chemical Equilibrium").

The order of the experiments has been altered also. The manual begins with a section on the structure of atoms and ends with a selection of experiments on some of the largest molecules known, the proteins. In the intermediate chapters we develop those topics that are treated in most general chemistry laboratory manuals—descriptive inorganic chemistry, the behavior of gases, thermodynamics, kinetics, chemical equilibrium and oxidation-reduction. The sequence in this manual conforms quite closely to that in a new general chemistry textbook, *Contemporary Chemistry* by John E. Hearst and James B. Ifft (W. H. Freeman and Company, in press), but the sequence of experiments can be easily altered to fit that of virtually any general chemistry text.

In addition to the reorganization, some important new material has been added. The section on atomic and molecular structure has been updated. It consists of the original experiment on crystal structure, from the previous editions, plus two new experiments that should complement today's increased emphasis on attempts to provide the correct quantum-mechanical description of the atom. A new experiment on qualitative analysis with the use of paper chromatography has been added to demonstrate some of the newer methods available for detecting the presence of ions in solution. For the many students interested in the biological sciences, two experiments in the field of biochemistry have been added to the section on organic chemistry.

A modest treatment of classical qualitative analysis has been preserved. The methods employed in this area of experimental chemistry illustrate the principles of chemical equilibrium. The five original experiments plus the two new experiments provide a sound foundation for an understanding of qualitative analysis.

This manual contains 39 experiments—approximately twice the number performed in the standard general chemistry course. The instructor can thus choose from an ample selection those experiments that will best complement the lecture portion of his course.

A large number of problems are presented in the individual study assignments, and in the exercises at the ends of the experiments. These problems present a challenge to the student's understanding of the chemical principles illustrated in the laboratory experiments. They vary in complexity from the completely straightforward problems (the first few in every grouping) to some that are quite difficult and for which the students may need to seek the help of the laboratory instructor.

This manual contains laboratory report forms. The advantages of these forms are that they provide explicit guidance on what data to tabulate and how to arrange them so that they can be treated expeditiously. In addition, the report forms show the student how to perform some of the more difficult calculations. For a few of the experiments, the student is required to provide his own report form. This exercise should help him to prepare for subsequent independent laboratory research.

Level. This laboratory manual is designed for an intermediate level course in general chemistry. By "intermediate," we mean a course suitable for students in the life and earth sciences, for those chemistry and physics students who have not had high-school chemistry,[1] and for students in the

[1]For students who have had a sound preparation in chemistry in high school, the more advanced manual in this series, *Chemical Principles in the Laboratory,* is recommended.

social sciences and humanities. This manual does not require mathematics beyond the second-year course in high-school algebra. If a student has difficulty with some of the calculations or graphical techniques, he will find Appendixes A and B helpful.

Acknowledgments. We acknowledge our gratitude to Harper W. Frantz and W. H. Freeman and Company for inviting us to be the continuing authors of this prestigious and highly successful series of laboratory manuals. Indeed, one of us (J. B. I.) initially learned the essentials of laboratory chemistry from the first edition of one of the manuals in this series. We continue to believe that this manual will provide a thorough foundation in the principles of the chemical laboratory. We trust that the current revision has retained the clarity and effectiveness of earlier editions, as well as introducing some recent experiments that will expose students to the important developments that are taking place in modern chemistry.

We are also grateful to our expert typist and friend, Eleanor Scott, who has so beautifully transformed our often confusing drafts into a typed manuscript ready for publication.

January, 1975

James B. Ifft
Julian L. Roberts, Jr.

FRANTZ/MALM'S
Essentials of Chemistry in the Laboratory

CONTENTS

INTRODUCTION

The chemistry laboratory can be a place of joy, discovery, and learning. It can also be a place of danger and frustration.

The danger arises from the nature of the chemicals and apparatus that are used in these experiments, and their effects on protein. Skin consists of protein. We hope that you do not have to experience firsthand the fact that even 6 *F* solutions of acids and bases, not to mention concentrated solutions, have a most adverse effect on protein. The eyes are especially sensitive to acids, bases, and oxidizing agents. In addition, the open flame of a bunsen burner presents a continual hazard to clothing and hair.

The frustration is generated by experiments that don't seem to work, or by data on your report form that seem unintelligible when you are attempting to do the calculations the night before the report is due. You can minimize both types of problems by careful recording of data and thoughtful consideration of the data while collecting it.

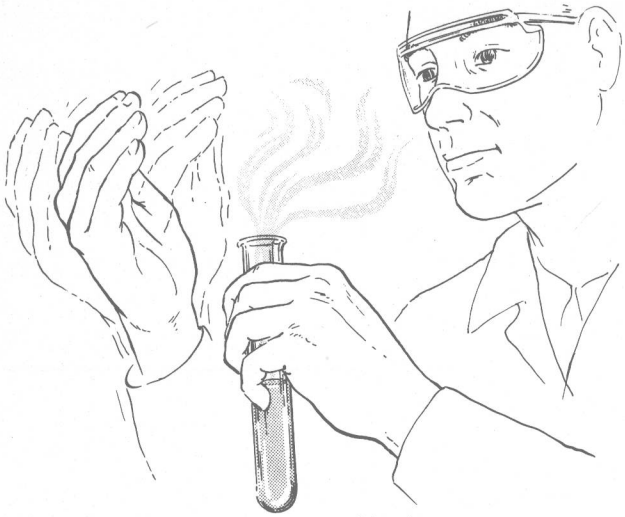
We strongly advise you to learn and observe at all times the following laboratory rules and regulations in order that you will minimize the potential dangers and frustrations of laboratory work, and maximize the joy.

SAFETY RULES

These rules are designed to ensure that all work done in the laboratory will be safe for you and your fellow students.

1. The most important safety rule is that glasses must be worn at all times in the laboratory. Prescription glasses are adequate in almost all situations. If you do not wear glasses, obtain an inexpensive pair of safety glasses from your chemistry stockroom. In some procedures—such as heating a crucible to dryness or evaporating an acid solution—be sure to wear safety goggles or to carry out the experiment in a hood, which is provided in most laboratories for this purpose.

If any chemical comes in contact with the eye, the most effective first aid is the immediate flushing



ALWAYS smell a substance by wafting its vapor gently toward your face.

FIGURE i-1
The procedure for smelling a substance.

of the eye with copious amounts of tap water. You are seldom more than a few seconds from a faucet. Continue flushing for at least five minutes and then consult a physician at once. If your laboratory is equipped with eye fountains, familiarize yourself with their use and their location.

2. Fire is a constantly present danger. Learn where the nearest fire extinguisher is and how to use it. Your laboratory should also be equipped with a safety shower or fountain: if your hair or clothing should catch on fire, go to it at once and douse yourself.

3. Minor burns, cuts and scratches are fairly common injuries. However, you must report every such incident to your instructor, who will determine what first aid is appropriate. If you or another student must report to the infirmary or hospital, be certain that someone else accompanies the injured person.

4. Bare feet are not allowed in a chemistry laboratory. Broken glass and spilled chemicals, such as concentrated acids, are all too common on the floors of chemistry labs. In addition, we recommend that bare legs, midriffs and arms be covered with old clothing or, preferably, with a laboratory apron or coat.

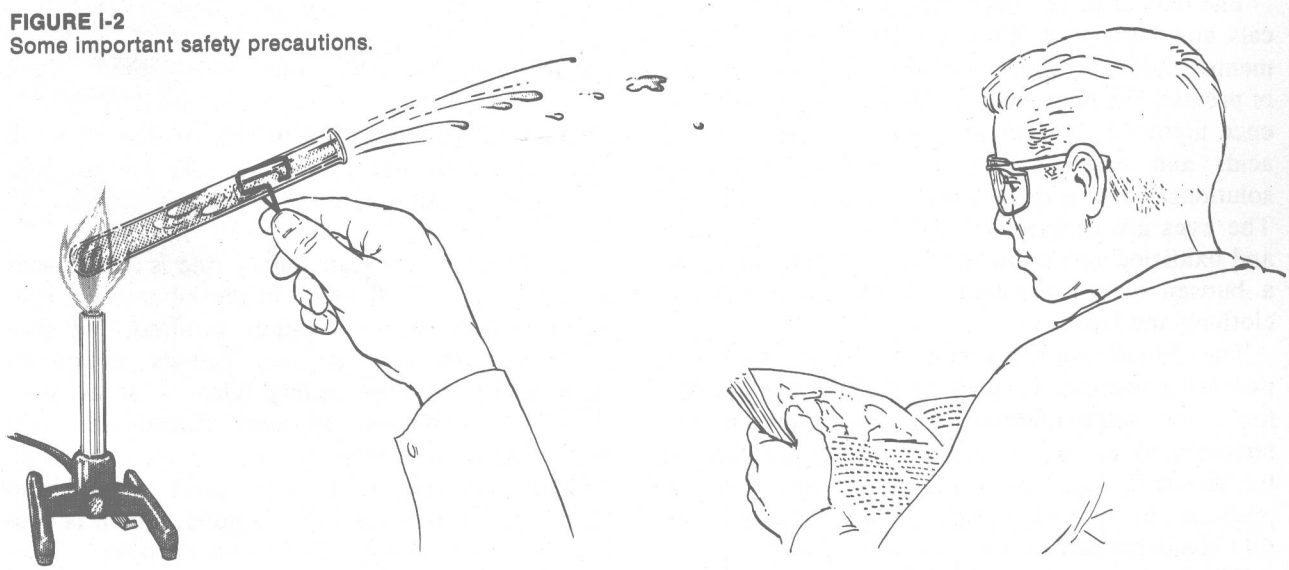
5. The vapors of a number of solutions are quite potent and can irritate or damage the mucous membranes of the nasal passages and the throat. Use the technique displayed in Figure i-1 when you need to sniff an odor.

6. In many experiments it is necessary to heat solutions in test tubes. Never apply heat to the *bottom* of the tube; always apply it to the point at which the solution is highest in the tube, working downward if necessary. Be extremely careful about the direction in which you point a tube; a suddenly formed bubble of vapor may suddenly eject the contents violently (an occurrence called "bumping"). Indeed, a test tube can become a miniature cannon.

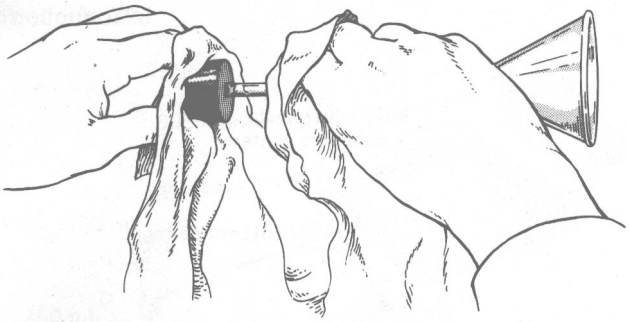
7. Taste chemicals and solutions *only* when directed to do so. (Poisonous substances are not always so labeled in the laboratory.)

8. Beware of hot glass tubing—it *looks* cool long before it may be handled safely.

FIGURE i-2
Some important safety precautions.



NEVER point a test tube of boiling liquid at your neighbor—it may bump.
SAFETY GOGGLES, worn regularly in the laboratory will protect your eyesight.



NEVER force a thistle tube or funnel into a stopper by grasping the large end. Use the stem and twist as you push. ALWAYS wrap your hands in a towel when putting a glass tube into a stopper. Moisten with water and insert with a twisting motion.

FIGURE i-3
The procedure for inserting a glass tube into a stopper.

9. For reactions involving poisonous gases, *use the hood,* which provides suction to remove such gases or vapors.

10. Neutralize spilled acid or base as follows: (a) Acid on clothing; use dilute sodium bicarbonate solution. (b) Base on clothing; use dilute acetic acid. (c) Acid or base on the desk; use solid sodium bicarbonate for either, followed by water.

11. To insert glass tubing (including thermometers, thistle tubes, etc.) through a rubber stopper, first lubricate the tube and stopper with water or glycerine. Hold the tubing with a cloth *near the end to be inserted,* and insert with a twisting motion. (If you twist a thistle tube by the "thistle" end, it is easily broken.)

LABORATORY REGULATIONS

These regulations are designed to guide you in developing efficient laboratory techniques and in making your laboratory a pleasant place to work.

1. You must read each experiment thoroughly before entering the lab. If you do not, you will waste a great deal of time (both your own and your instructor's), you may expose yourself and others to unnecessary hazards, and you will probably not obtain reliable, useful data. (You will also routinely fail all pre-lab quizzes if your instructor chooses to use them.)

2. Discard solids into the waste crocks. *Never throw matches, litmus, or any insoluble solids* into the sink. Wash down liquids into the sink with much water; acids and salts of copper, silver, and mercury are corrosive to lead plumbing.

3. Leave reagent bottles at the side shelves. Bring test tubes or beakers *to the shelf* for transferring chemicals and carrying them to your desk.

4. Read the label *twice* before taking anything from a bottle.

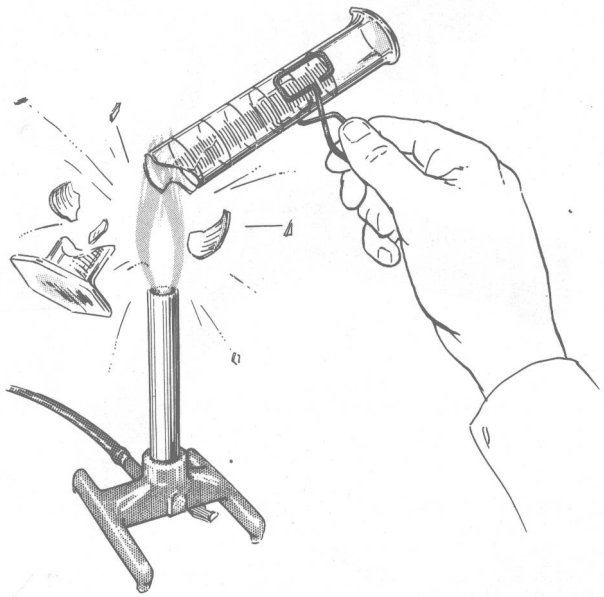
5. Avoid using excessive amounts of reagent—1 to 3 ml is usually ample for test tube reactions.

6. *Never* return unused chemicals to the stock bottle. You may make a mistake from which other students' experiments will suffer.

7. Do not insert your own pipets or medicine droppers into the reagent bottles. Avoid contamination of the stock solution by pouring the solution from the bottle.

8. Do not lay the stopper of a bottle down. Impurities may be picked up and thus contaminate the solution when the stopper is returned. Hold the stopper as illustrated in Experiment 2, Figures 2-3 and 2-4.

9. Do not heat heavy glassware such as volumetric flasks, graduated cylinders, or bottles; they break easily and heating distorts the glass so that the calibrations are no longer valid. Test tubes may break if they are heated above the liquid level and liquid is then splashed over the hot glass. Evaporating dishes and crucibles may be heated red hot. Avoid heating any apparatus too suddenly; apply the flame intermittently at first.



NEVER heat a graduated cylinder or bottle

FIGURE i-4
If heat is applied to the wrong type of laboratory apparatus, the outcome can be disastrous.

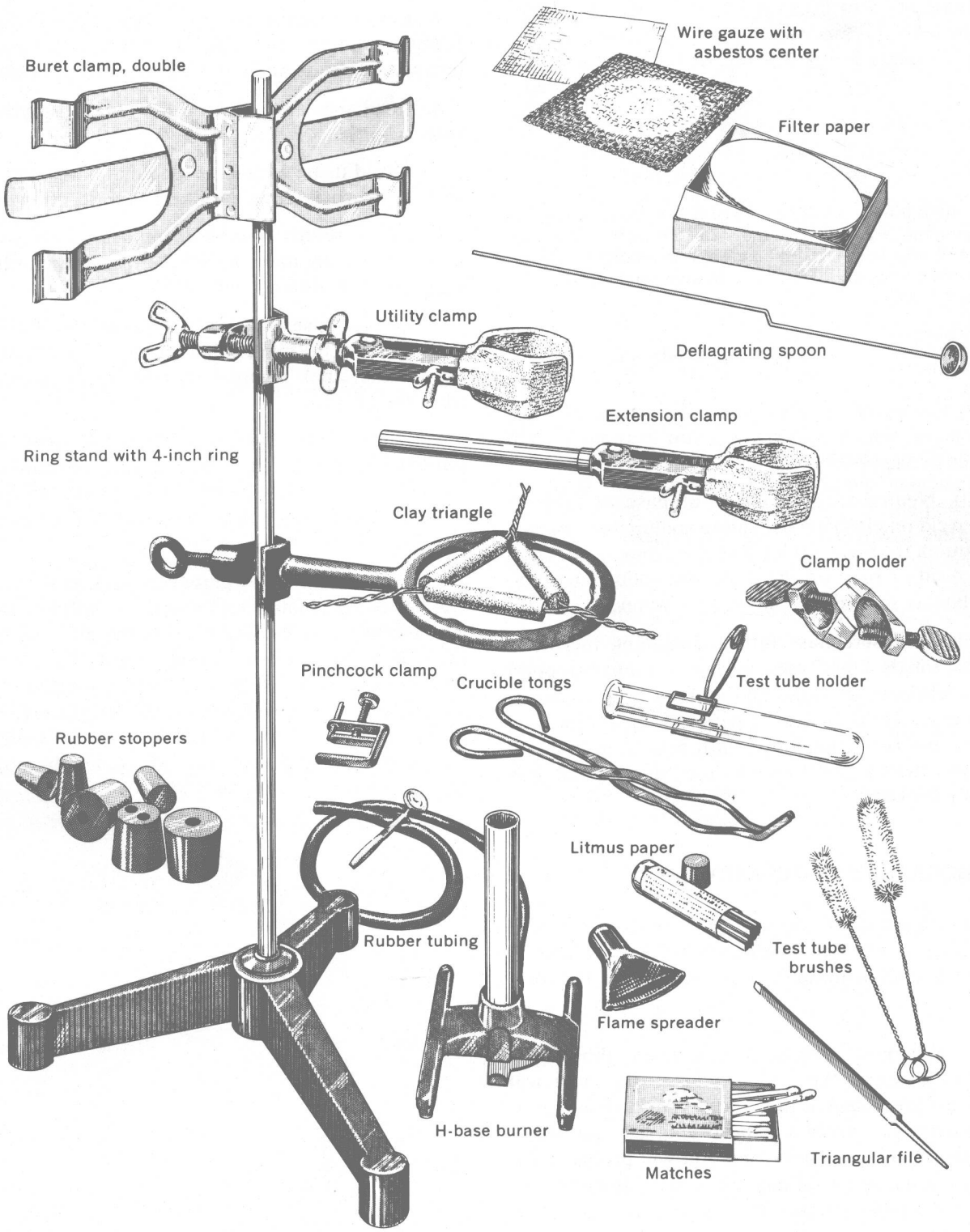


FIGURE i-5
Common Laboratory equipment. [From J. W. Hagen, *Empirical Chemistry*, W. H. Freeman and Company. Copyright © 1972.]

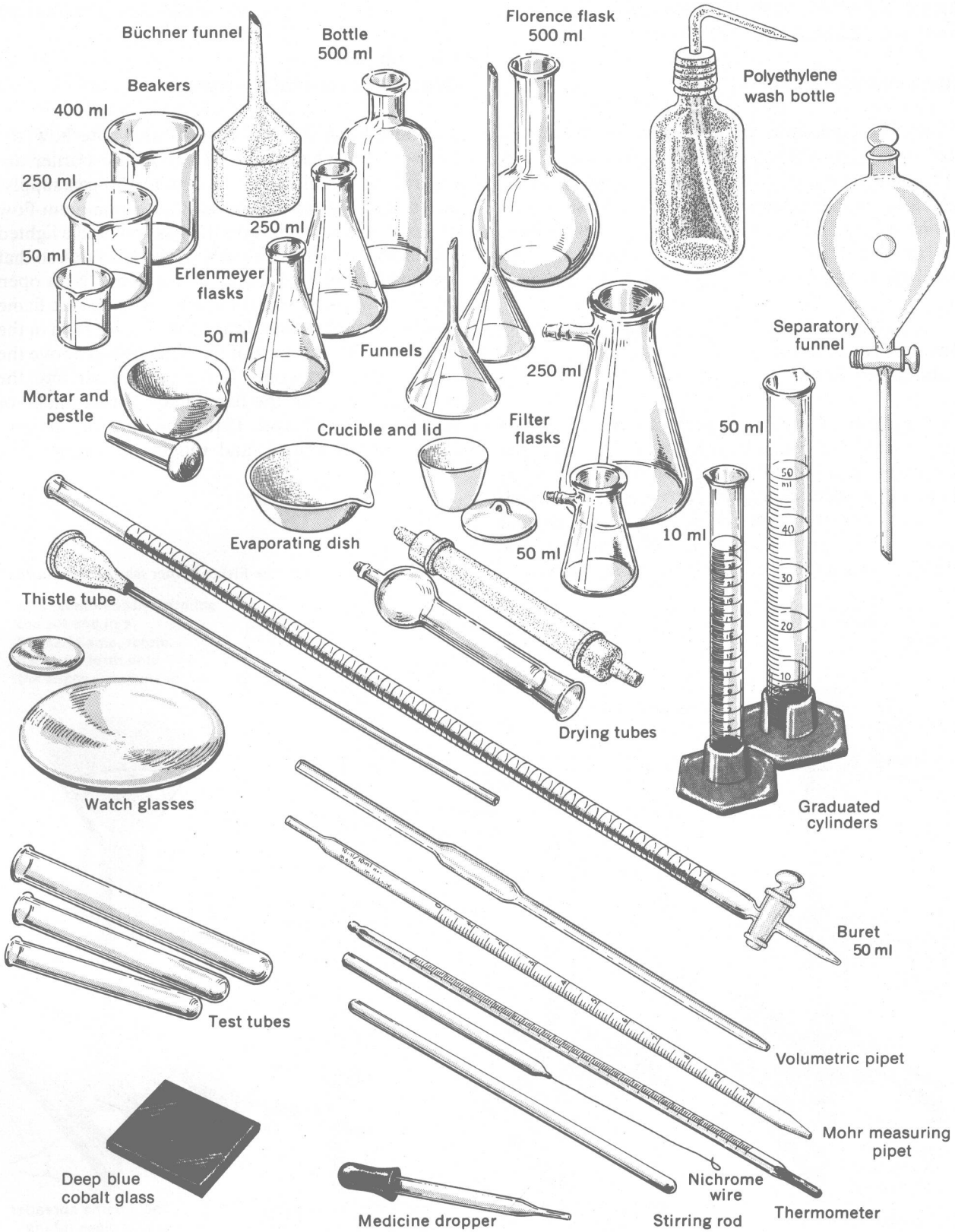


FIGURE i-5 (continued)