INTRODUCTORY CHEMISTRY LABORATORY MANUAL

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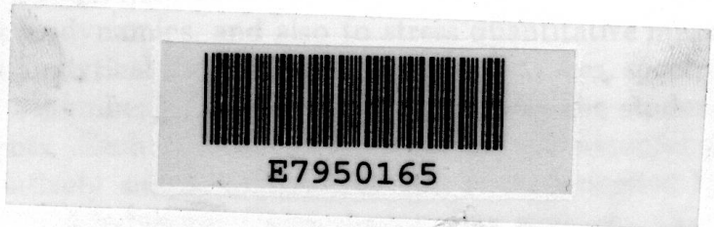

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Introductory Chemistry Laboratory Manual

Chemistry - Laboratory manuals

A SERIES OF BOOKS IN CHEMISTRY

James W. Cobble, Editor

Preface

This manual is intended to provide a set of challenging laboratory experiments to accompany any up-to-date general chemistry text. The experiments have been chosen to demonstrate principles that are emphasized in modern textbooks and to develop skills in basic experimental procedures. The experiments stress quantitative measurements, and unknowns have been introduced wherever possible.

For some years now laboratory texts have been available which significantly upgrade the content of high school chemistry laboratories, with the result that many college freshmen find the general chemistry laboratory a duplication of their high school laboratory, or at best an inadequate challenge to their ability. At the same time, there have been significant changes in college chemistry, notably the introduction of thermodynamics and quantum mechanics in moderate depth in the general chemistry course, and the phasing out of the traditional sophomore analytical chemistry course. All these changes have put pressure for improvement on the general chemistry laboratory, which has resulted in the introduction of new experiments in some laboratories, and the appearance in many general chemistry laboratories, in ever-increasing numbers, of such instruments as single pan balances, spectrophotometers, and pH meters.

In recognition of the changes occurring in both high school and college chemistry, the experiments in this manual have been chosen to illustrate principles of descriptive chemistry and elementary thermodynamics, and also to stress quantitative measurements and application of quantitative analytical procedures. Analytical balances, spectrophotometers, and pH meters are used in a number of experiments to develop the students' skill in using these important instruments. Each new laboratory skill or instrumental procedure is normally introduced in a relatively simple experiment and is then applied in a different or more demanding manner in a subsequent experiment. For example, the titration technique is introduced in Experiment 2 by means of an acid–base indicator titration, then applied in following experiments to oxidation—reduction and complexometric, pH, and potentiometric titrations.

Although I feel that the student benefits from writing research-style reports on the experiments, not everyone agrees that the benefit to the students offsets the resulting heavy grading demands on the instructor. In an effort to provide a more flexible reporting

procedure, instructions are given for writing formal reports, but each experiment is also provided with a summary report form which may be used in place of, or in addition to, a research-style notebook, according to the preference of the instructor.

To help the student prepare for each experiment, advance reading recommendations are provided for a number of contemporary textbooks. Three textbooks are referred to:

Dillard, C. R., and D. E. Goldberg, *Chemistry: Reactions, Structure, and Properties* (New York: Macmillan, Inc., 1971).

Mahan, B. H., *University Chemistry* (Reading, Mass.: Addison-Wesley, 1969), 2nd ed.

Sienko, M. J., and R. A. Plane, *Chemistry: Principles and Properties* (New York: McGraw-Hill, 1966).

I am deeply grateful to Professors J. Aaron Bertrand and Peter E. Sturrock for their many helpful suggestions and criticisms during the writing of this manual. I would also like to acknowledge the assistance of Mr. Gerald O'Brien, who designed the dc power supply and the student ammeter described in Appendix B.

H. R. H.

Contents

Introduction

LABORATORY CONDUCT AND PROCEDURE

General Rules and Suggestions

You are in the laboratory primarily to learn by performing experiments. You will benefit most from the experience if you have prepared in advance by studying the assigned material in the laboratory manual and in any recommended references. It is particularly useful for you to mentally review the sequence of operations to be performed and to recognize the ones that are time consuming or hazardous so that you may decide on the proper course of action.

Do not depend on other students or the instructor to provide you with information you should obtain for yourself. The instructor will recommend reference sources when asked, but it is your responsibility to seek independently the information you need.

Keep in mind at all times that you share the laboratory and certain items of equipment with other students. Common sense dictates that each student should perform his work in a safe and courteous manner. Loud noises, practical jokes, and similar offensive conduct have no place in the chemistry laboratory.

Do not attempt to repair or adjust malfunctioning instruments. Report them immediately to your instructor so that he may make arrangements for their repair.

When you leave the laboratory, your working area must be clean and in good order. This applies to your workbench, reagent shelf, sink, and areas shared with other students. Waste materials should be disposed of in the manner described by your instructor. Normally, receptacles are provided for insoluble solids; soluble chemicals are discarded by flushing them down the sink with water.

Safety

The success of industrial safety awards programs in reducing bodily injury accidents clearly indicates that accidents can be prevented by publicizing and enforcing safety regulations. Similarly, accidents in the chemistry laboratory can and must be prevented. It is your responsibility to follow the general safety rules and to look out for potentially hazardous operations so that you may learn how to perform them safely.

Learn in advance the location of fire blankets, fire extinguishers, safety showers, eyewash fountains, and other emergency equipment so that they may be used without delay if the need arises.

If you do not wear glasses, you are required to wear safety glasses whenever you are in the laboratory. Removable side shields are recommended for use with either regular or safety glasses.

Laboratory aprons may be purchased to protect yourself and your clothing against spilled chemicals. The use of aprons is strongly recommended.

Most chemicals are poisonous to some extent when taken internally, and many are poisonous or capable of causing painful burns when spilled on the skin. For this reason you should not intentionally taste any laboratory chemical, and a rubber bulb should be used when pipetting to prevent accidental introduction of chemicals into your mouth. For the same reason, eating, drinking, and smoking in the laboratory are forbidden.

Glass tubing should not be used until the cut ends have been fire-polished to round the sharp edges. When inserting glass tubing or thermometers into rubber stoppers, the glass should be well lubricated with glycerin or water, and the glass should be grasped near the end to be inserted to minimize the risk of its breaking. A worthwhile precaution is to use a towel between the glass and your hands so that in the event of breakage you will be protected from cuts.

Never add water to concentrated sulfuric acid, since the heat of dilution may cause it to splatter. Instead, you should dilute the acid by adding it slowly with stirring to a large amount of water. Should you need to neutralize *any* relatively concentrated acid or base, this also should be done cautiously and with ample stirring so that the heat of neutralization does not cause splattering.

When you discard a soluble chemical in the sink, it should be slowly added to a steady stream of water from the tap, and the water should be left running long enough to ensure that the chemical has been thoroughly flushed down the drain. This will prevent inflammable or poisonous substances from collecting in the traps, as well as preventing the reaction between chemicals discarded in succession.

First Aid

It is your responsibility to know the location of, and how to use, the emergency equipment.

If you splash acids, bases, or other chemicals on your person or in your eyes, *immediately* wash the affected area with *large quantities* of water, giving *first attention* to your *eyes.* Continue washing until all corrosive materials have been washed away. The eyelid should be pulled up and away from the eye, by another person if necessary, while the eye is thoroughly irrigated with *plain* water. An eyewash fountain, eyewash bottle, or water fountain may be used for this purpose. When this has been done, report to your instructor.

In the event of cuts, burns, and the like, report to your instructor at once.

THE NOTEBOOK AND THE REPORT

Rules for Keeping the Notebook

The laboratory notebook is used to make an original, permanent record of your work, and consequently a *permanently bound* notebook is required. Some general rules for using your notebook correctly are given below. Your instructor will inform you in more detail concerning the type of notebook required, and the practices to be followed in its use.

1. The first two or three pages of the notebook should be left blank, so that they may be used to prepare an index. The index should be added to and kept up to date as each report is completed. If the notebook pages are not numbered, you should number them consecutively, in ink.
2. All experimental observations should be recorded directly in the notebook, in ink, at the time of observation. It is very poor practice to record figures on loose scraps of paper to be later copied neatly into the notebook. Such scraps of paper tend to be easily lost, but a more important reason for condemning this practice is that it destroys the value of the notebook as the *original* record of your laboratory work.
3. Do not obliterate or erase any data recorded in the notebook. If you wish to correct an error, draw a single line through the incorrect number or statement, and then write the correct entry beside it. If the reason for the change is not obvious, a brief explanation should be written in parentheses.
4. Do not remove pages from the notebook.
5. Try to be neat. Neatness can generally be improved by studying the experiment and thinking it through in advance. This will help you decide on the clearest and most efficient way of recording your observations, and also to make any necessary advance preparations, such as preparing a table to be filled in at the laboratory. Some students strive for neatness by writing their formal reports using only the right (or left) side of the notebook pages, leaving the other side for sketches and calculations.

Writing the Report

The report for each experiment should contain each of the sections listed below, arranged in the order presented.

1. The TITLE of the experiment.
2. The PURPOSE of doing the experiment. Here you should list the skills to be learned or the principles to be demonstrated by properly performing the experiment.
3. The PROCEDURE followed. *Briefly* describe the apparatus used and the procedure followed. It is not necessary to list the minor details of the procedure unless a new procedure has been substituted for the one given in the laboratory manual.
4. RESULTS. In this section you are to record the actual experimental observations, such as the original and final buret readings in a titration experiment, or the appearance of a precipitate in a qualitative analysis experiment.
5. DISCUSSION. In this section you use your experimental observations to perform calculations or to draw conclusions. You should clearly show exactly how you performed the calculations, rather than just presenting the final results.
6. QUESTIONS AND PROBLEMS. Many of the experiments in this laboratory manual have a number of questions or problems at the end of the experiment. These should be answered in the notebook as part of the report.

Since you have a limited amount of time in the laboratory in which to complete each experiment, as much of the report as practical should be written outside the laboratory. The Title, Purpose, and Procedure sections may be written before the laboratory period. However, all experimental observations must be recorded in the notebook *only* at the time they are made. The Discussion and Questions and Problems sections may be completed either in the laboratory or at home, depending on the time available.

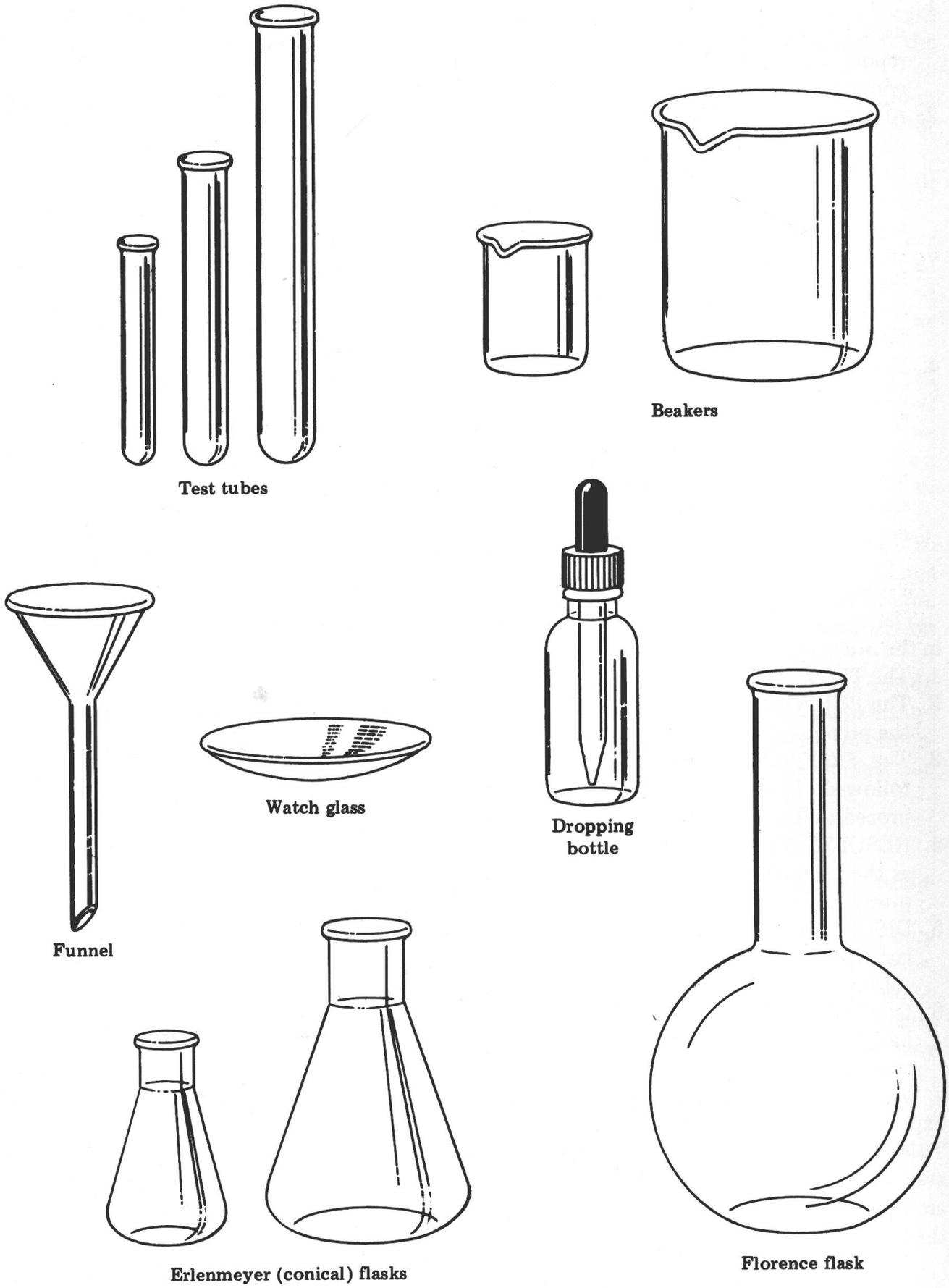


Figure I-1. General glassware.

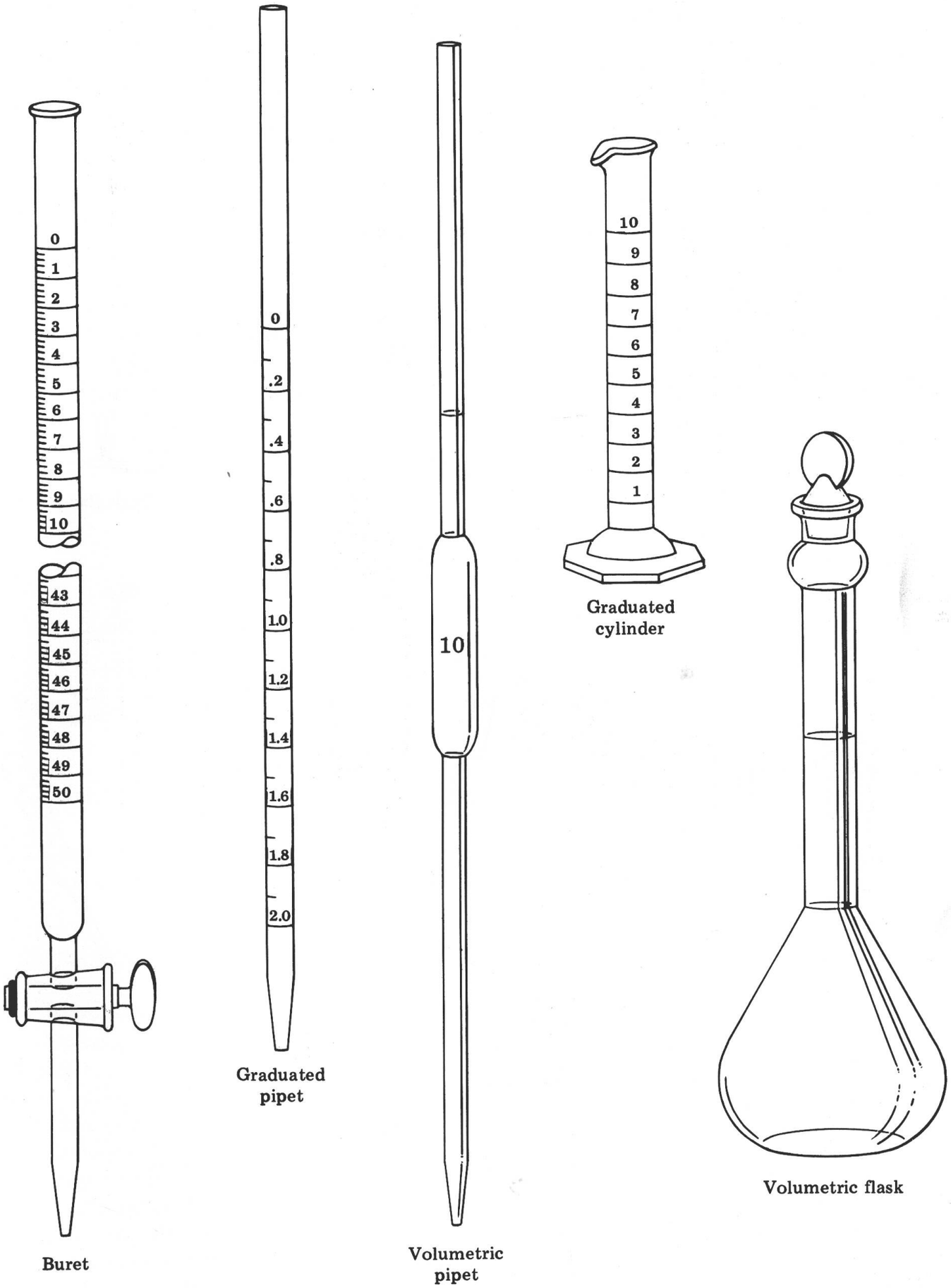


Figure I-2. Volumetric glassware.

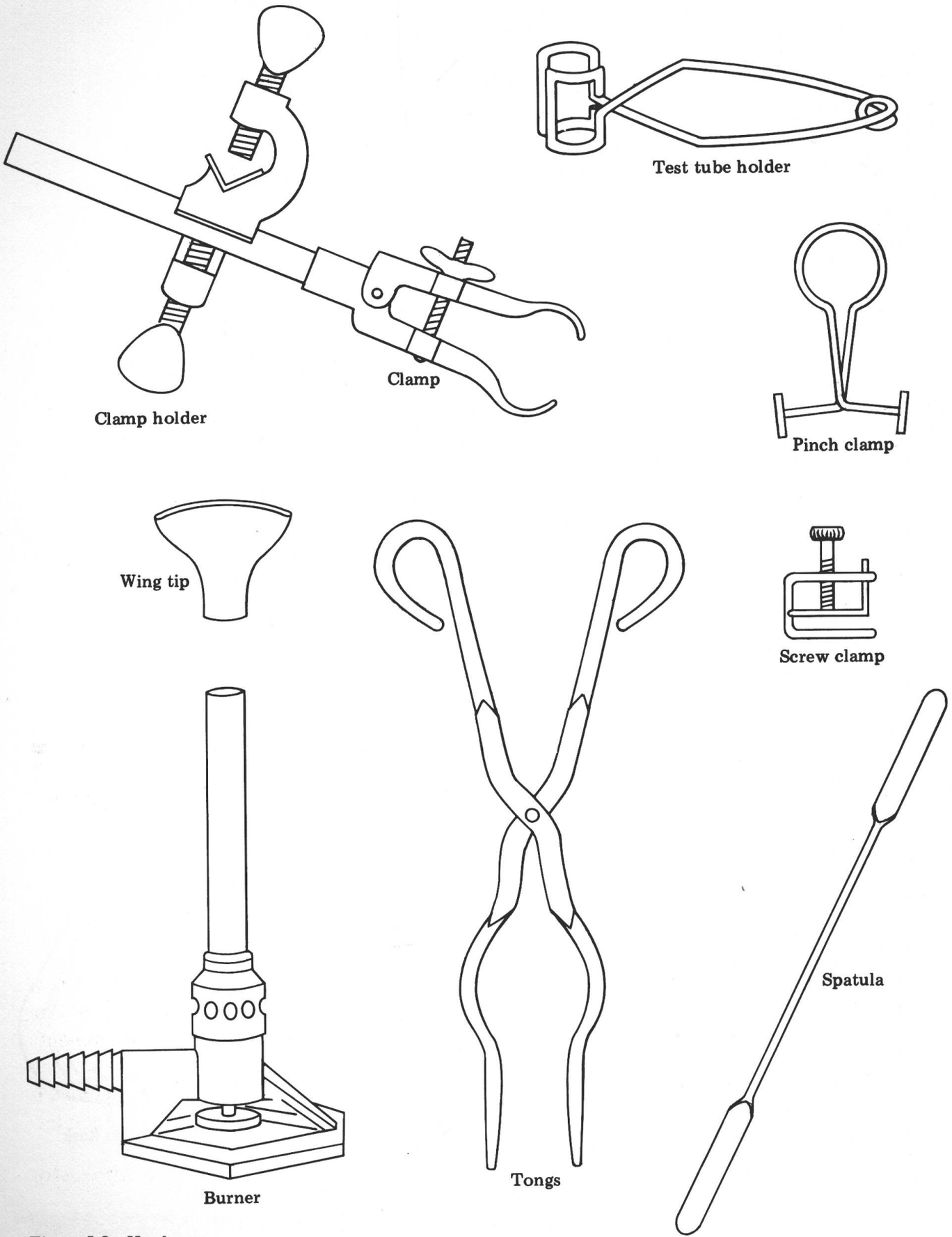


Figure I-3. Hardware.

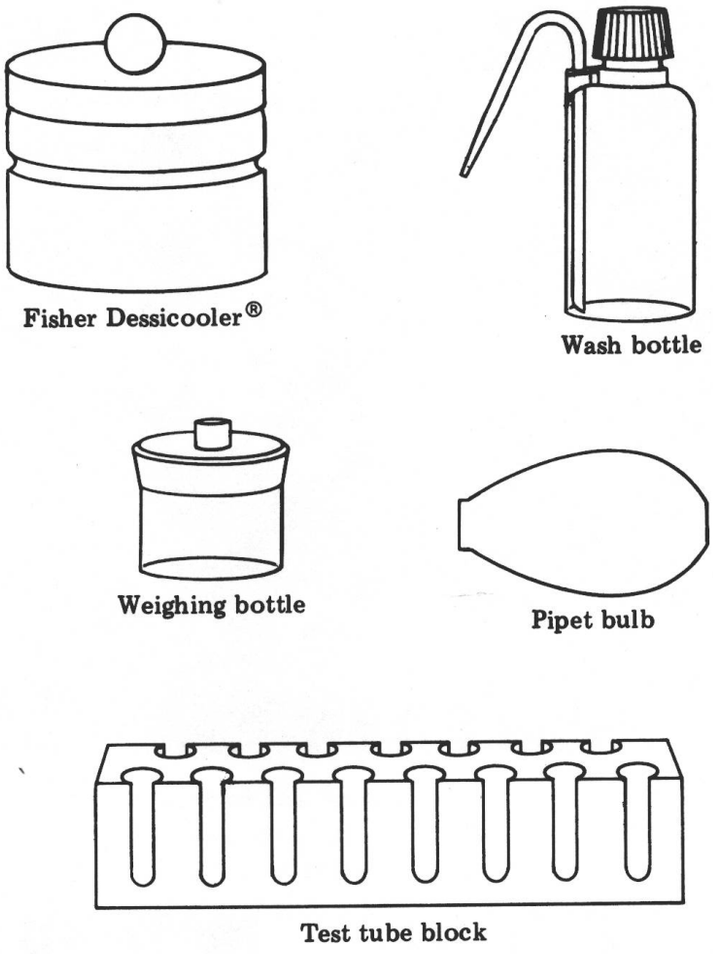


Figure I-4. Miscellaneous equipment.

LABORATORY EQUIPMENT

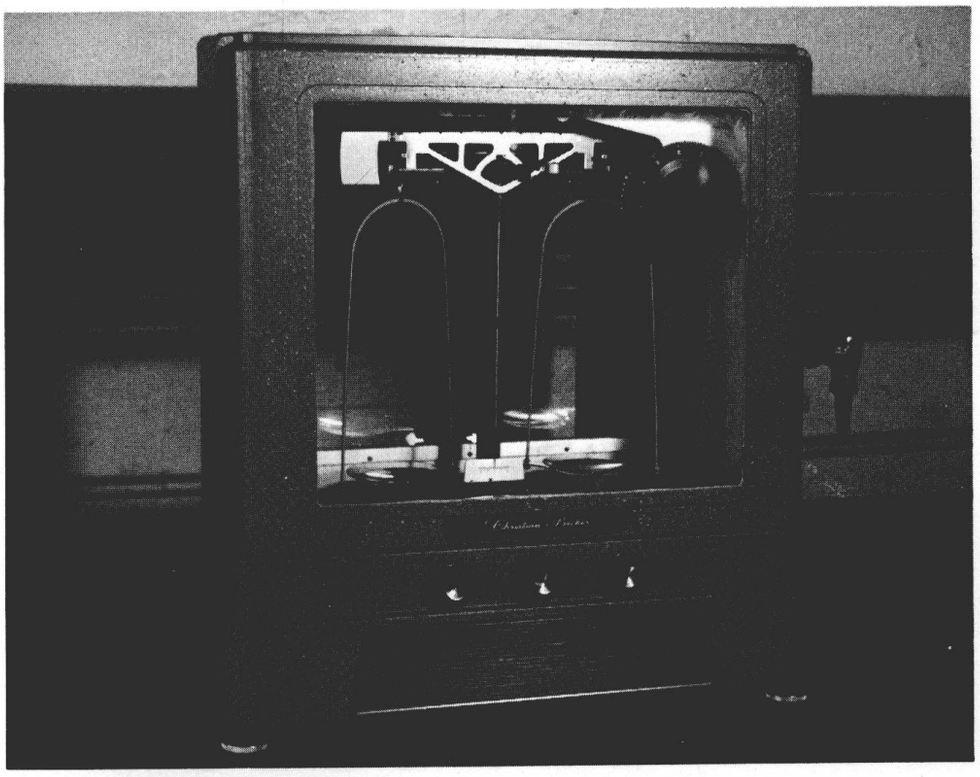
Common items of laboratory equipment are shown in Figures I-1 through I-4. Study the figures until you are able to identify any unfamiliar items.

THE BALANCE AND WEIGHING

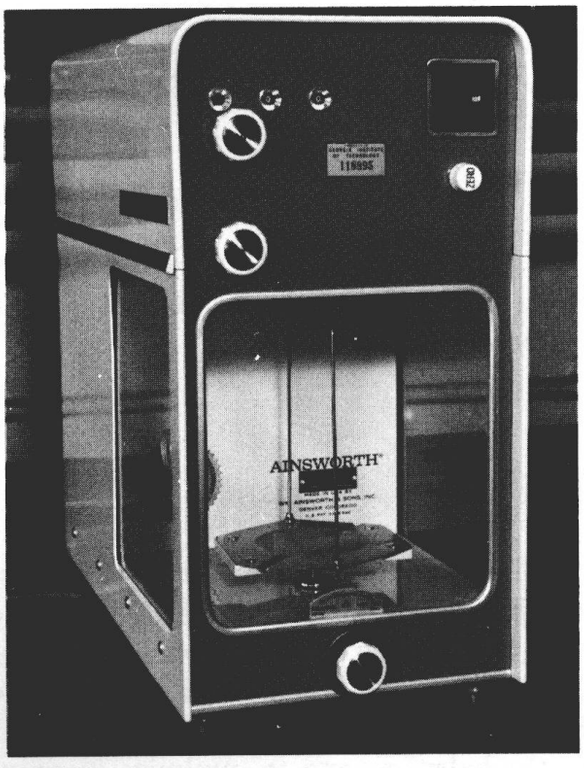
Rules for Using the Analytical Balance

Two types of analytical balances are currently widely used in chemistry laboratories: equal arm balances (identified by two pans), and substitution balances (identified by one pan). These instruments are illustrated in Figure I-5. Both types are precision instruments capable of weighing to the nearest 0.1 mg (0.0001 g). Like most delicate instruments, the balances contain parts (bearings, weights, etc.) which are subject to damage if the balance is improperly used. Therefore, to prevent damage to the balances, and to minimize inconvenience to other students, you are asked to read and obey the following rules.

1. Do not attempt to repair or adjust a malfunctioning balance. Inform the instructor immediately, and he will see to it that the problem is corrected.
2. Never place chemicals on the balance pans. Solids should be weighed on a previously weighed piece of paper or watch glass; liquids should be weighed *only* in a stoppered container, such as a flask or weighing bottle.



An equal arm balance.



Ainsworth

Mettler

Substitution balances.

Figure I-5. Examples of laboratory analytical balances.