SIENKO AND PLANE

EXPERIMENTAL CHEMISTRY

SECOND EDITION

GRAW-HILL BOOK COMPANY, INC.

Michell J. Sienko
Professor of Chemistry
Cornell University

Robert A. Plane
Associate Professor of Chemistry
Cornell University

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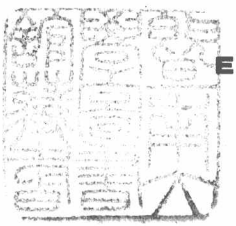


EXPERIMENTAL CHEMISTRY

PREFACE

THIS LABORATORY MANUAL has been written to accompany our textbook "Chemistry." The chief purpose in writing it is to present to the student a course in experimental chemistry that brings out the distinction between observation and interpretation. Most students, even those with good high-school preparation in chemistry, have a rather hazy notion of the distinction between an observed fact and an explanation of that fact. Therefore, we have tried to emphasize with these experiments that the chemical laboratory is a place where one learns chemistry by seeing what substances do under fixed conditions. We offer few revolutionary experiments. Most of them have already been suggested in one form or another by the host of chemistry teachers who have preceded us and to whom we owe an immeasurable debt. What we do offer is a change from "Demonstrate that this is true" to "Find out what happens." We emphasize quantitative experiments.

The manual is separated into four parts. Part I describes the techniques of experimental chemistry, including safety precautions, methods of recording data and results, gravimetric and volumetric operations, glass manipulation, the generation and handling of gases, and the use of a simple slide rule. Some of these techniques are described so that they can be used as assigned experiments should the time be available and should the instructor find it advisable. Individual sections of Part I are prefixed with a *T*.

Part II presents a selected set of experiments (with prefix *E*) designed to teach the important principles and facts of general chemistry. We have tried to keep the individual experiments directed toward a clearly stated purpose without introducing many interesting but confusing side issues. In later experiments, we have attempted to put the student more and more on his own. With the exception of steps that could be dangerous, the procedural directions become progressively more concise and the data sheets progressively less complete. Throughout the manual, text references are given only where pertinent. Included in Part II are four problem sessions. In our experience at Cornell we have found it extremely helpful to use such problem-solving sessions to replace some of the laboratory periods. Students are sent to the blackboard to work out by themselves an interrelated set of problems while the instructor offers individual guidance. In the absence of blackboard space, the same thing can be done at the laboratory bench.

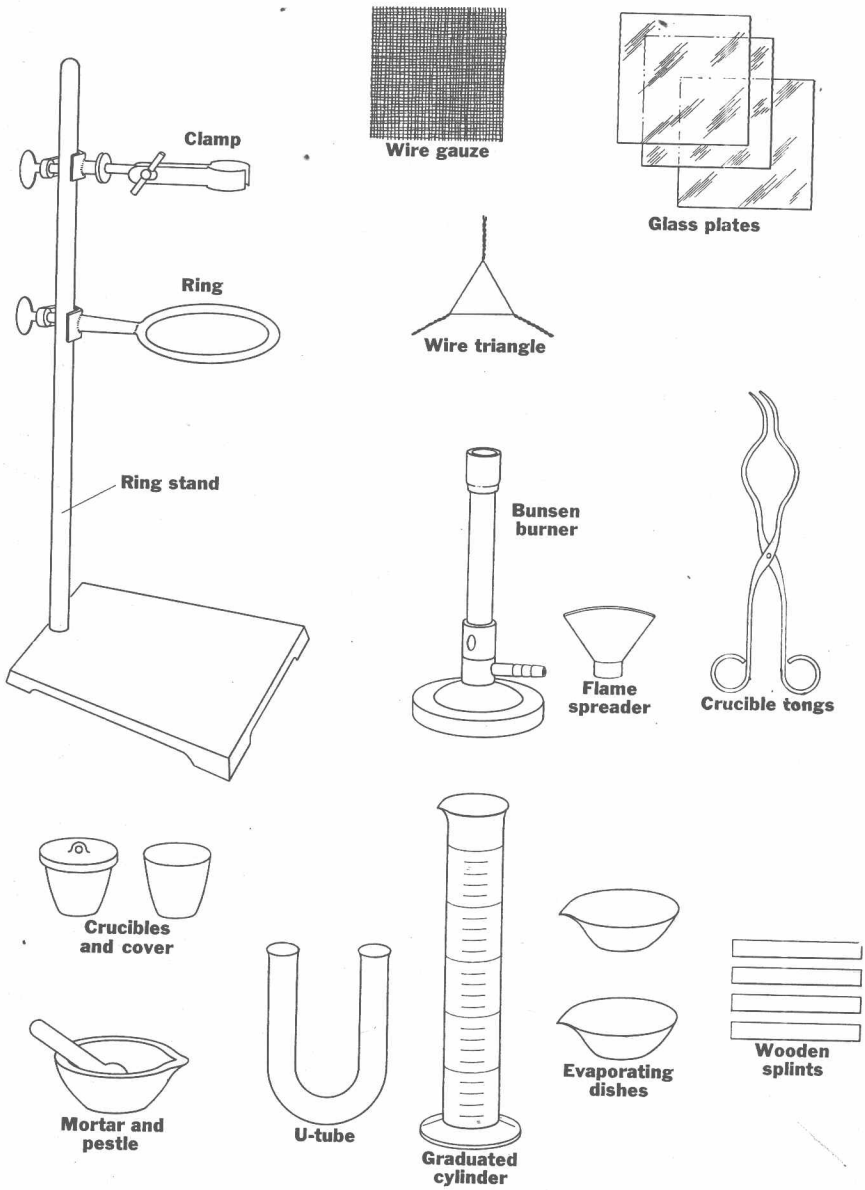
Part III (prefix *Q*) is a somewhat abbreviated scheme of qualitative analysis based on use of thioacetamide in which elements are taken up and added in the order of the periodic table. As each new element is added, the student investigates

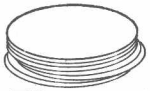
its chemistry so as to find out how it behaves without his knowing what should happen. Thus, he seems to devise his own scheme.

Part IV contains general-reference material that will be of use to students in the laboratory. Reference material relating to single experiments is found within the experiments. Information useful to instructors is found in an instructor's manual available from the publisher.

MICHELL J. SIENKO
ROBERT A. PLANE

Typical Chemistry Laboratory Equipment

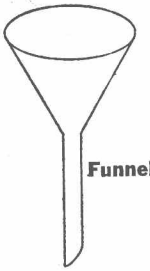




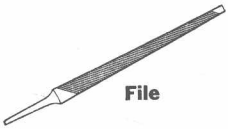
Filter papers

Rubber stoppers

Cork



Funnel



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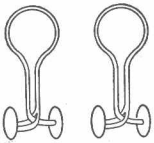
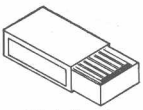
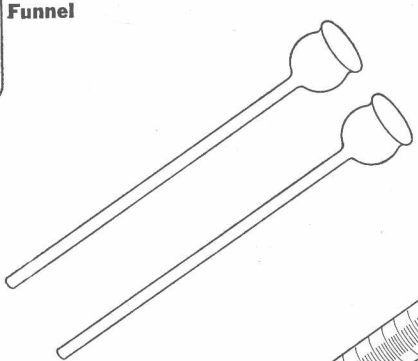


Pinch clamps

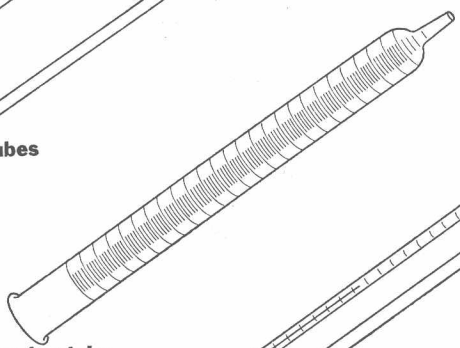
Rubber connectors



Matches

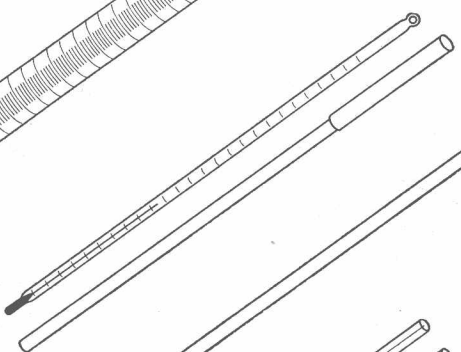


Thistle tubes



Measuring tube

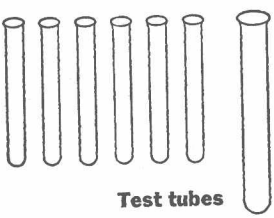
Test tube holder



Thermometer and case

Glass rod (1')

Capillary tubes



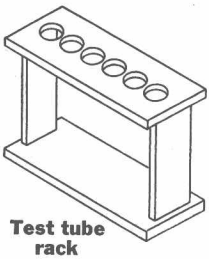
Test tubes

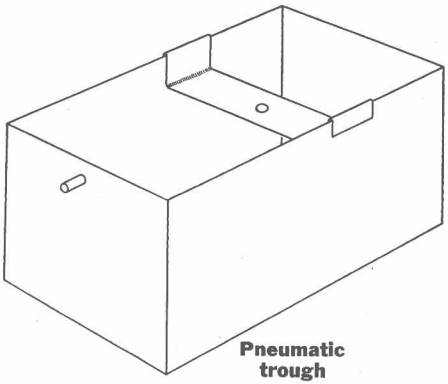
Watch glass

Medicine dropper

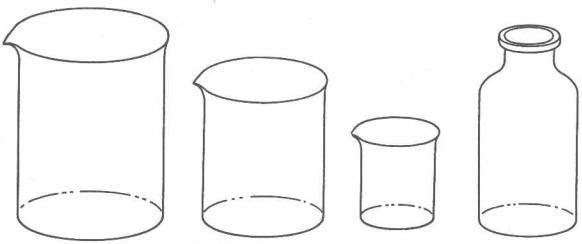

Asbestos board

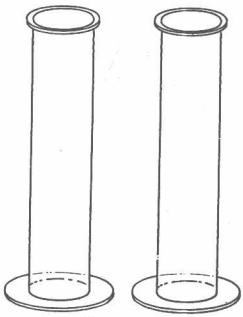

Test tube brush

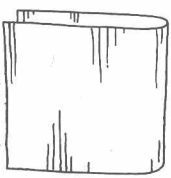

Test tube rack

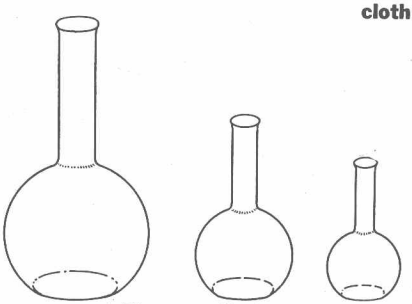

Pneumatic trough

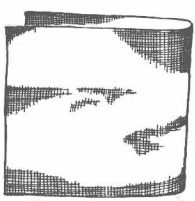

Rubber tubes


Beakers
Bottle


Plain cylinders


Desk cloth


Florence flasks
Leveling bulb


Cheese cloth

EXPERIMENTAL CHEMISTRY

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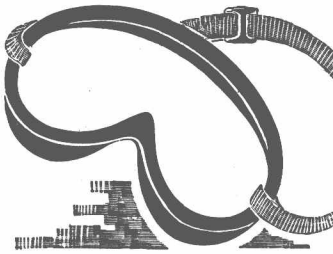


T1: SAFETY

THE CHEMISTRY LABORATORY is actually not a dangerous place, but it demands a reasonable prudence on the part of an experimenter to keep it a safe place. In the following paragraphs, the more important precautions are discussed:

Eye protection. The eyes are particularly susceptible to permanent damage by corrosive chemicals as well as by flying fragments. It is strongly recommended that each student equip himself with a pair of safety glasses (if he does not ordinarily wear prescription glasses) and that he *wear these all the time he is in the laboratory.* In doing hazardous experiments, follow all directions carefully and, in particular, take care not to endanger your neighbor. For example, when heating a test tube never point its mouth toward anyone. Report any accident immediately to your instructor. All injuries involving the eyes should be referred at once to a physician.

Cuts and burns. The great majority of laboratory injuries are cuts and burns. Practically all these can be prevented by following a few simple rules:

(1) Never try to insert glass tubing, especially thistle tubes, into rubber stoppers without first moistening the tubing and the hole. Also, it is a wise precaution to shield the hands by use of cheesecloth, as shown in Fig. T1.1.

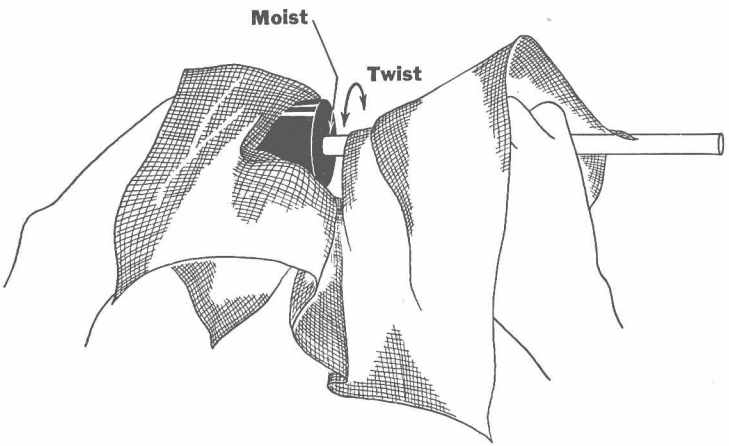


Fig. T1.1

To reduce leverage on the glass, *hold the hands close together.* While twisting the stopper back and forth, gradually work the glass tubing through the hole. It may be necessary to moisten the stem several times while the operation progresses.

(2) Fire-polish all sharp edges of broken glass. This operation takes but a few seconds (see Sec. T6), but it may save your hand.

(3) When trying to remove glass tubing from a stopper or from a rubber fitting, lubricate it by prying a bit of the rubber away from the glass and dribbling in a bit of water. Then try to twist off the stopper. It may be necessary to relubricate. If the joint is really stuck, do not force it. Cut off the rubber with a razor blade.

(4) If a glass stopper sticks in a bottle, gently tap the stopper with a piece of wood (e.g., with a test-tube rack). If it still sticks, call your instructor.

(5) Never pick up a heated piece of glass until it has had time to cool. Unfortunately, hot glass looks just like cold glass; so, when in doubt, use tongs.

(6) Use of glass equipment that is cracked or broken may ruin an experiment. Worse, it may cause an injury. Since damaged equipment needs to be replaced eventually, discard it immediately.

(7) In case of any injury, report at once to your instructor for treatment.

Poisonous chemicals. Most of the chemicals you will work with are poisonous to some degree. It is obvious that you should never taste a chemical unless specifically directed to do so. However, there are more subtle ways of being poisoned. One of these is breathing in toxic vapors. Be careful to work in a ventilating hood whenever instructed to do so. Even such common substances as carbon tetrachloride, benzene, and mercury are poisonous and potentially dangerous. Avoid prolonged exposure to these liquids or the accompanying vapors. Since heating favors the vapor state, these and other poisonous liquids should be heated only in a hood.

Occasionally, you will be directed to test the odor of a substance. The proper way to do this is to waft a bit of the vapor toward your nose as shown in Fig. T1.2. Do not inhale the vapor directly from the test tube.

A possible poisoning hazard, frequently overlooked, is contamination through the hands. Some poisons—e.g., benzene—are rapidly absorbed through the skin. All poisons can stick to the hands and eventually end up in the mouth. Always scrub your hands thoroughly after exposure to

Fig. T1.2

hazardous chemicals, and get into the habit of always washing your hands before leaving the laboratory.

Essential precautions. Follow all directions with utmost care, especially those having to do with hazardous conditions. Do not perform any unauthorized experiment. If you want to change or supplement the assigned material, first consult your instructor and get his permission. Irresponsible behavior will result in immediate expulsion from the laboratory.

In using chemical reagents, double-check the label to make sure you are not using the wrong chemical. Serious explosions have frequently resulted from such errors.

Smoking is not permitted in the laboratory.

T2: RECORDING RESULTS

THE TRUE RESEARCH CHEMIST considers his notebook as one of his most valuable possessions. It summarizes all the work that he has done and the results he has obtained. Loss of a notebook can be catastrophic. Furthermore, the notebook record must be kept so that it will be meaningful at a later date both to its author and to other chemists.

In this course, you are asked to keep your record in the laboratory

manual. Data should be recorded as soon as they are obtained. Consult with your instructor as to choice between pen or pencil. Writing should be concise and legible. Qualitative data, which are just as important as quantitative data, should not be omitted. A few words of description will often suffice if their meaning is clear. In recording quantitative data, make sure the numbers are labeled both as to units and as to property being measured. Pay strict attention to significant figures (see text, Sec. 1.3) so that the numbers give maximum information.

In calculating results from experimental data, it is good practice to show specifically the method of calculation. This can be done either by giving a sample calculation using numbers labeled with units or by writing out a general expression indicating how the experimental data are mathematically combined. For example, in an experiment to determine density, we can write either

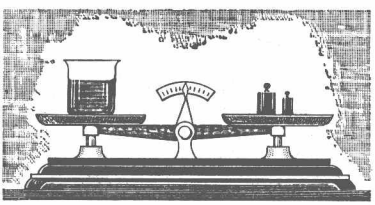
$$\text{Density of unknown} = \frac{27.1 \text{ g.}}{22.6 \text{ ml.}} = 1.20 \text{ g./ml.}$$

or

$$= \frac{\text{mass of unknown}}{\text{volume of unknown}}$$

In neither case is it useful to show the detailed arithmetic, which is better done on scratch paper or in some inconspicuous place in the record book.

You will note that for the earlier experiments in this manual labeled blanks are provided to indicate the particular data needed for the experiment. However, in later experiments this is not done. You will have to decide for yourself what experimental observations need to be made and recorded. If in doubt, consult your instructor in order not to overlook an essential measurement.



T3: WEIGHING

ONE OF THE MOST common operations in experimental chemistry is the determination of mass or weight (see Sec. 1.5 in the text for the distinction between these terms). The mass of an unknown is usually determined by comparing its weight with the weight of a known mass. For making the comparison, there are two kinds of balance in common use. One of these is the platform balance, two examples of which are shown in Fig. T3.1. In using the type shown on the left, the unknown on the left pan is balanced