Stanley Marcus
The Late Michell J. Sienko
Robert A. Plane

Experimental General Chemistry

实验普通化学〔英〕

McGraw-Hill Book Company
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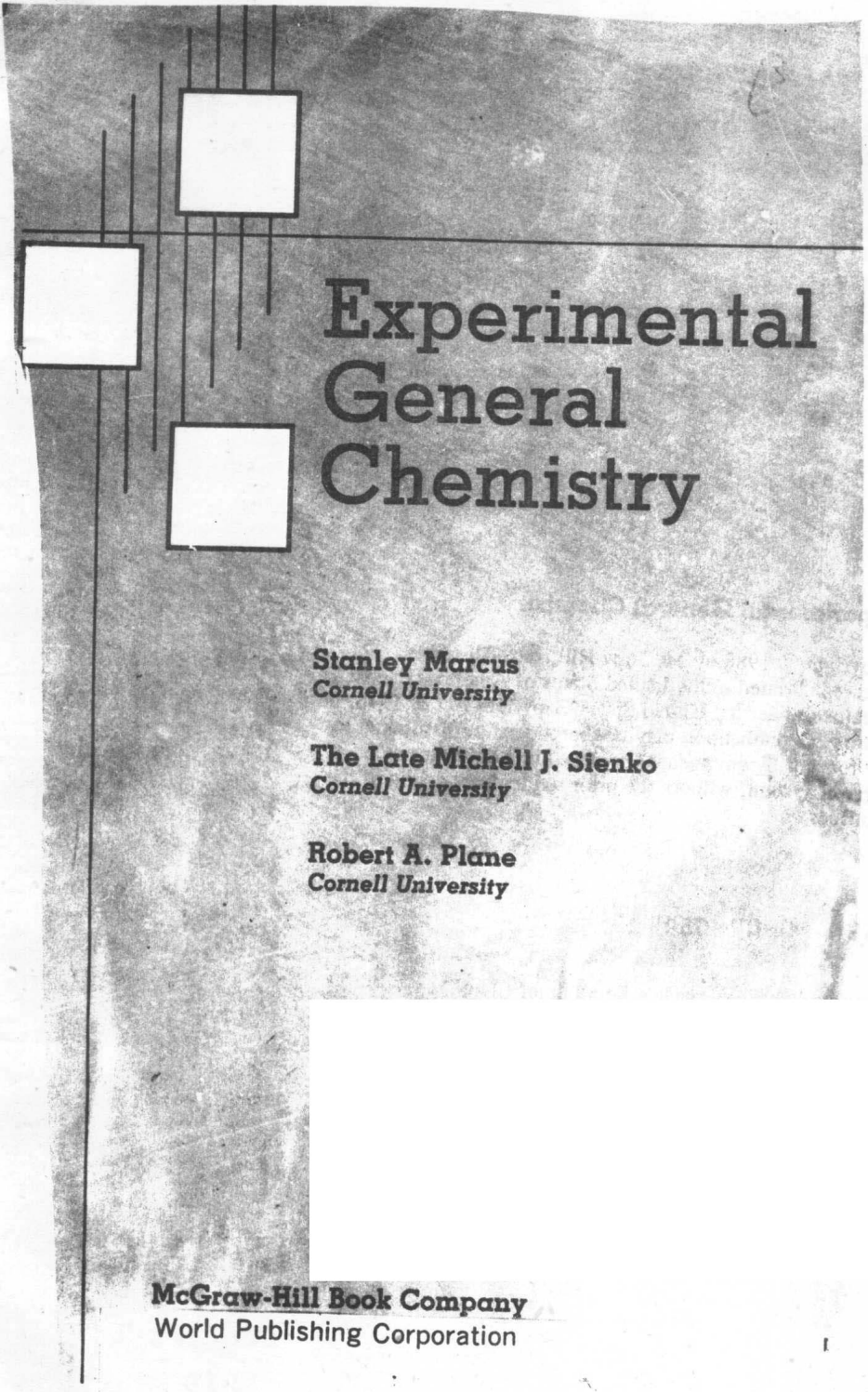

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Preface

This laboratory manual can be used with any general chemistry textbook, including books designed for courses that cover qualitative analysis and/or a substantial amount of organic chemistry. Most of what is contained in this manual has been drawn from the laboratory manual *Experimental Chemistry, Sixth Edition*, by Sienko, Plane, and Marcus. The experiments all have been tested by thousands of students, both at Cornell University and at many other schools.

The two main differences between this manual and *Experimental Chemistry* are

1. The numbering of the experiments has been changed in order to group experiments dealing with similar conceptual material together and to present them in the order followed by most general chemistry courses.
2. The number of organic chemistry experiments has been substantially increased.

The approach in this manual is *guided discovery* rather than simple verification. Students are taught to observe and to report what actually happens, not what they think should happen. Nearly all of the experiments are designed to be completed in a single two- or three-hour laboratory session, but there are also open-ended experiments that require several laboratory periods for completion. Experiments Q.1 through Q.10 (qualitative analysis), for example, require ten or more laboratory sessions. Experiments E.21 (Preparation of a Complex Iron Salt) and E.22 (Analysis of a Complex Iron Salt) could be done either as separate experiments or as a single combined experiment. Experiment E.34 (Separation and Analysis of a Mixture), which also uses procedures described in Experiments E.35 through E.37, could take as many as five periods to complete.

The manual has four major parts. Part A describes techniques of experimental chemistry. As is the case throughout the manual, the primary emphasis is on safety. Also discussed in this section are balances, volumetric measuring devices,

filtration techniques, the gas burner, glassworking, the centrifuge, determination of melting point, spectrophotometers, and proper methods of recording and treating data.

Part B presents 52 experiments designed to teach the important general principles and facts of general chemistry. For each major conceptual area of chemistry, there are several experiments that are at different levels of complexity. There are, for example, five experiments relating to properties of matter and Dalton's elementary atomic theory, five on the gas laws, five on atomic and molecular structure and bonding, more than fifteen on stoichiometry (synthesis and analysis), more than ten on solutions, three on kinetics, more than six on aqueous equilibrium, seven on thermodynamics and electrochemistry, and more than ten on descriptive chemistry.

Part C consists of eleven organic chemistry experiments. There are two molecular model exercises designed to familiarize the student with various types of isomerism and to provide some practice in naming organic compounds. Important classes of organic compounds (hydrocarbons, alkyl halides and alcohols, aldehydes and ketones, carboxylic acids and esters) are studied in a series of experiments by examining characteristic reactions of each functional group. There are also experiments on the synthesis of aspirin and sulfanilamide and on the paper chromatography of amino acids.

Part D is an abbreviated scheme of qualitative analysis in which groups of elements are added to the scheme not in the order in which they appear in the classical scheme of qualitative analysis but in the order in which they appear in the periodic table. As each element is added, the student carries out experiments to find out how that element fits into the scheme. The experiments are designed to allow the student to discover what eventually turns out to be the classical scheme of analysis. The main purpose of working through the series of experiments is to learn some of the chemistry of individual elements and some important principles of aqueous solution chemistry.

There are also five appendixes containing such general reference material as constants and conversion factors, equilibrium constants, and atomic weights, which will be of use to students in making computations.

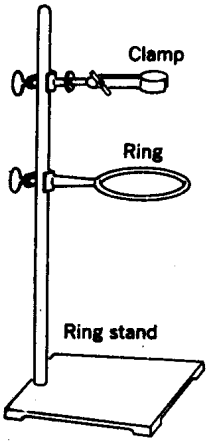
Detailed information on the preparation of reagents for the experiments and on answers to the questions is available in an instructor's manual, which can be obtained by writing to the publisher.

Most of the credit for this laboratory manual must be given to Michell J. Sienko, late professor of chemistry at Cor-

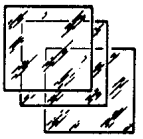
nell University, who developed the majority of the experiments. By his teaching, example, and writings, Dr. Sienko, together with his colleague Robert A. Plane, made an impact on chemical education in the United States and many other nations that may never be duplicated.

Stanley T. Marcus

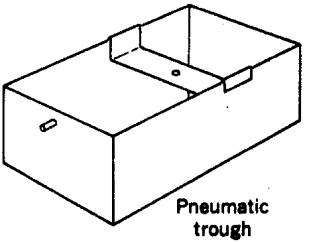
typical chemistry laboratory equipment

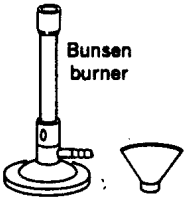



Wire gauze

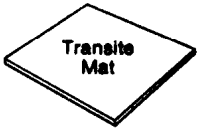

Glass plates


Test-tube brush


Pneumatic trough



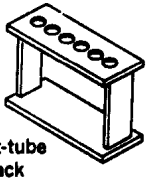
Flame spreader




Crucibles and covers

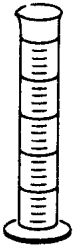

Wire triangle


Crucible tongs

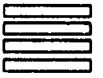

Test-tube rack

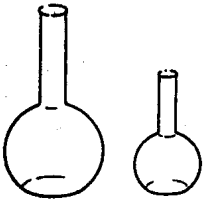

Mortar and pestle


U tube

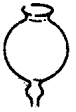

Graduated cylinder


Evaporating dishes

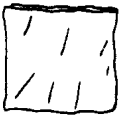

Wooden splints


Florence flasks


Erlenmeyer flask

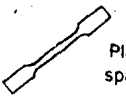

Leveling bulb


Cheesecloth

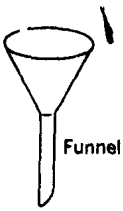

Desk cloth

Rubber stoppers

Cork

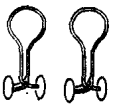


Plastic spatula

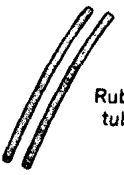


Funnel

File



Pinch clamps

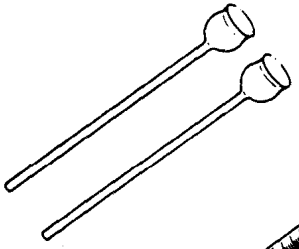


Rubber tubes

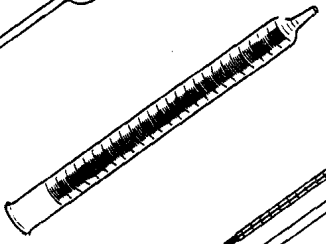
Filter papers

Matches

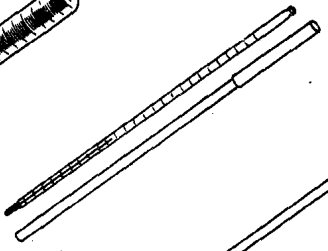
Rubber connectors



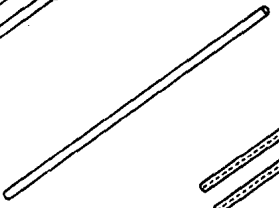
Thistle tubes



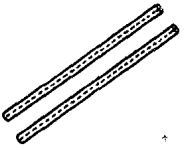
Measuring tube



Thermometer and case

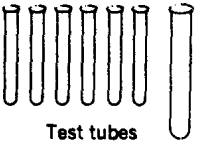


Glass rod (30 cm)



Capillary tubes

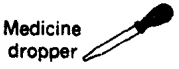
Test-tube holder



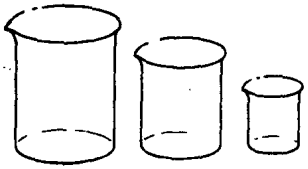
Test tubes

Watch glass

Rubber policeman

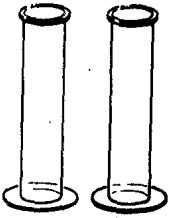


Medicine dropper



Beakers

Bottle



Plain cylinders

Typical Inventory

- 1 transite board
- 5 beakers: 30, 50, 100, 200, 600 mL
- 1 bottle, wide-mouth: ½ liter
- 1 bunsen burner with flame spreader
- 1 capillary: 25 cm
- 1 clamp
- 1 cheesecloth
- 2 crucibles and covers: No. 00
- 1 crucible tongs
- 1 cylinder, graduated: 25 mL
- 3 cylinders, plain: 125 mL
- 1 evaporating dish: No. 00
- 1 file
- 25 filter papers: 9 cm
- 3 flasks, flat-bottom: 125, 300, 500 mL
- 1 funnel: 65 mm
- 1 gas-measuring tube: 50 mL†
- 3 glass plates: 10 × 10 cm
- 1 leveling bulb
- matches
- 1 medicine dropper
- 2 pinch clamps
- 1 pneumatic trough
- 1 ring: 8 cm
- 1 ring stand: 45 cm
- 4 rubber connectors: 5 cm
- 1 rubber policeman
- 9 rubber stoppers:
  - 3 No. 8, 2-hole
  - 1 No. 5, 2-hole
  - 1 No. 4, 2-hole
  - 1 No. 4, 1-hole
  - 3 No. 1, 1-hole
- 60-cm rubber tubing: 7 mm
- 60-cm rubber tubing: 5 mm
- 15-cm ruler
- 10-cm spatula
- 10 splints
- 1 stirring rod: 5 mm × 30 cm
- 6 test tubes: 16 × 150 mm
- 1 test tube: 25 × 200 mm
- 1 test-tube brush
- 1 test-tube holder
- 1 test-tube rack
- 1 thermometer: −10 to 110°C
- 1 thistle tube
- 1 triangle
- 1 U tube
- 1 washcloth
- 1 watch glass: 80 mm
- 1 wire gauze: 10 × 10 cm

If qualitative experiments are included, each student should be provided with an individual set of reagents in 60-mL dropping bottles. These could profitably include: 6 *M* HCl, 15 *M* NH_3, 1.7 *M* thioacetamide, 3 *M* H_2SO_4, 3 *M* $NH_4C_2H_3O_2$, 2 *M* NaOH, 6 *M* NH_4Cl, saturated Na_2SO_4, 2 *M* $NaHSO_4$, 6 *M* HNO_3. Students also need a 15-cm piece of nichrome wire and a double thickness of cobalt glass. It is also helpful to have 12 additional test tubes (13 × 100 mm).

†Purchasable as "Dennis buret" or "Cornell buret" from Ace Glass Co., Vineland, N.J.

Contents

PART A

Techniques and Procedures

The entire structure of chemistry rests on a foundation of empirical evidence. To maintain the integrity of that foundation, chemistry insists that its facts be reproducible and independent of the observer.

In performing experiments, there are two absolute requirements: first, that the experiment not expose the experimenter or others to danger; second, that the experiment be designed and carried out such that valid information is generated.

The purpose of this part of the manual is to instruct you in practices and techniques that will lead to a safe working environment as well as to the generation of valid data.

1 Safety

The chemistry laboratory is really not a dangerous place, but it demands reasonable prudence on the part of an experimenter to keep it safe. 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 required that each student wear safety goggles at all times 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, do not point its mouth toward anyone. Report any accident immediately to your instructor. In case of injury to the eye, *immediately* flood the eye with lots of water, and continue to rinse for at least 10 min. If an eyewash fountain is not immediately available, use a rubber tube connected to a faucet. *All* injuries involving the eyes should be referred to a physician at once.

Cuts and Burns

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

1. In case of any injury, report it at once to your instructor for treatment.
2. Do not insert glass tubing, especially thistle tubes, into rubber stoppers without first moistening the tubing and the hole with water or glycerin. Also, it is a wise precaution to shield the hands by use of cloth, as shown in Figure A.1. To reduce leverage on the glass, hold the hands close together. While twisting the stopper back and forth, gradually work the glass

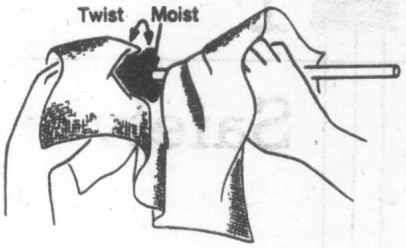


Figure A.1

tubing through the hole. It may be necessary to moisten the stem several times while the operation progresses.

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 or glycerine. Then try to twist off the stopper. It may be necessary to re-lubricate. If the joint is really stuck, do not force it. Cut off the rubber with a razor blade.

4 Never set heated glass on the bench top. Place it on a transite board to cool.

5 Never pick up a heated piece of glass until it has had time to cool. Unfortunately, hot glass looks just like cold glass; 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 must be replaced eventually, discard it immediately. Place it in a "waste glass" container, not in the wastepaper basket.

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 by breathing 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

Figure A.2

vapor toward your nose as shown in Figure A.2. Do not stick your nose in and inhale 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. Immediately scrub your hands thoroughly after exposure to hazardous chemicals, and get into the habit of always washing your hands before leaving the laboratory.

Food should not be brought into the laboratory.

Corrosive Chemicals on Skin or Clothing

If you should accidently spill a corrosive chemical, such as a concentrated acid or base, on yourself, quickly remove any contaminated clothing, flush the affected skin for at least 10 min with water, and notify your instructor. Do *NOT* neutralize chemicals on the skin by adding other chemicals.

Footwear

Shoes, not sandals, should be worn in the laboratory. Preferably, the shoes should be of the hard-toe variety.

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 or her 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.