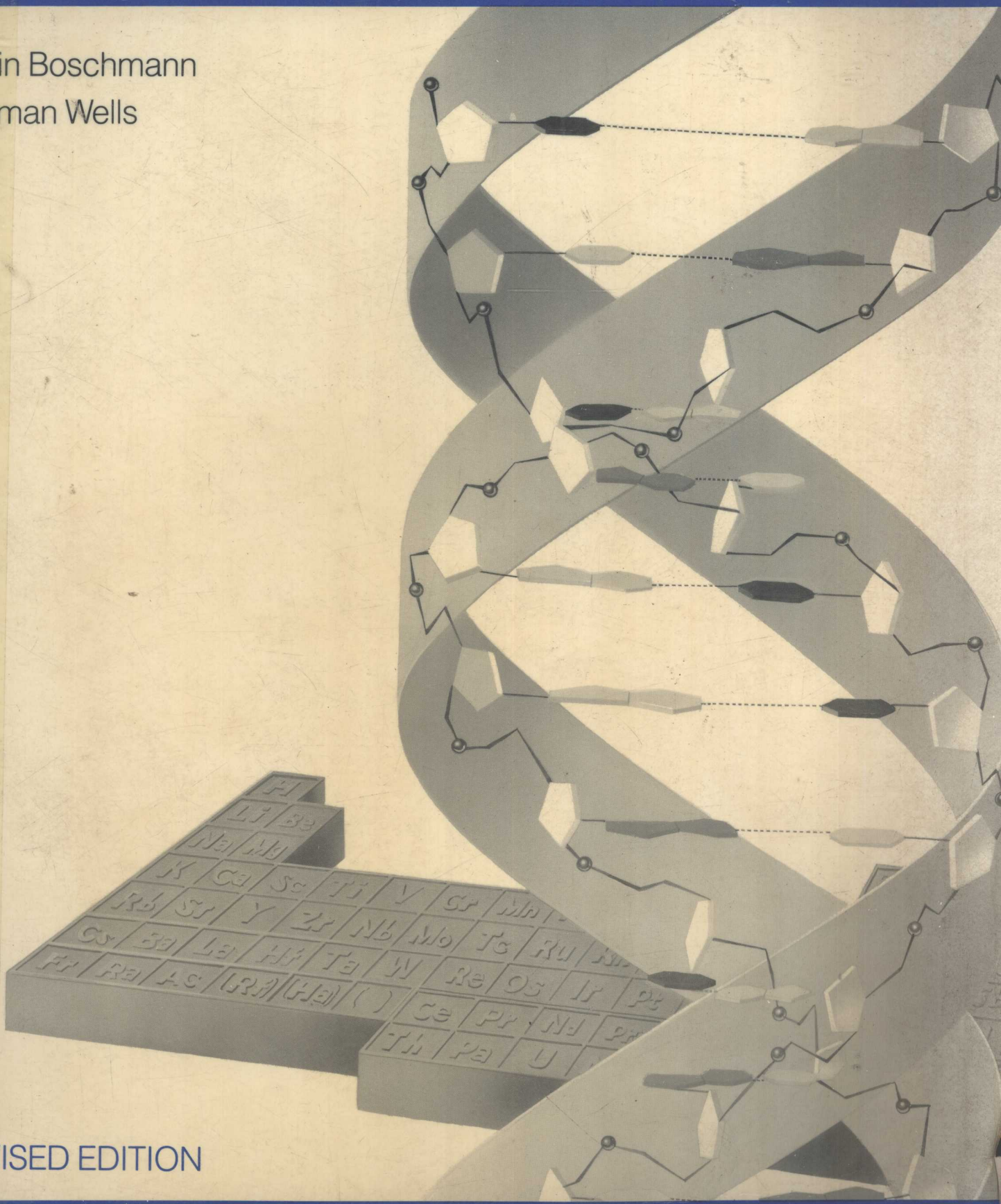
CHEMISTRY IN ACTION

A LABORATORY MANUAL FOR GENERAL, ORGANIC, AND BIOLOGICAL CHEMISTRY

Erwin Boschmann
Norman Wells



REVISED EDITION

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A Laboratory Manual for General, Organic, and Biological Chemistry

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ERWIN BOSCHMANN

Indiana University-Purdue University at Indianapolis

NORMAN WELLS

St. Lawrence University

McGRAW-HILL BOOK COMPANY

New York St. Louis San Francisco Auckland Bogotá Hamburg Johannesburg London Madrid Mexico Montreal New Delhi Panama Paris São Paulo Singapore Sidney Tokyo Toronto

This book was set in Press Roman by Automated Composition Service, Inc.
The editors were Stephen Zlotnick and Sibyl Golden;
the cover was designed by Joan E. O'Connor.
The cover illustration was done by George Kanelous.
Project supervision was done by The Total Book.
Semline, Inc., was printer and binder.

Artwork in Experiments 23 and 24 by Lisa Hite-Wadler.

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Organic, and Biological Chemistry

1234567890SEMSEM8987654

ISBN 0-07-006529-2

PREFACE

Theory guides, but experiment decides! Nowhere is this more true than in the sciences. Until a theory has been put to rigorous experimental tests, it cannot hope to advance to the status of law. The scientific method depends on a curious mind, careful and unbiased observation, and the ability to generalize. This manual provides an opportunity to develop all three aspects of the method. Most of us learn much by seeing and hearing, but (fortunately) we learn even more by doing. Experimentation provides a superb opportunity to allow lecture material to become real. Lavoisier is widely referred to as the father of chemistry, largely because he introduced experimentation using careful measurements which allowed valid conclusions.

It is the aim of this manual to provide a setting for discovery by observing, measuring, doing, using instrumentation, collecting data, drawing conclusions. The experiments are written for the nonscience major, beginning chemistry student. They illustrate basic chemical principles and are arranged in the approximate order of topics found in most current texts. The early experiments consider observations and measurement. Later the mole concept is brought in, with organic and biochemical experiments coming toward the end.

The experiments were developed over a 5-year period. They vary in content from the strictly academic exercise to the examination of commercial products and the study of industrial laboratory processes. Some are classical ideas; many are either truly original, or novel approaches to traditional designs. We had help from some 5000 students who tested and suggested, colleagues in the professional and industrial fields who stimulated and contributed, and dozens of educators from across the country who challenged and improved.

The nonorganic experiments can be completed within a 2-hour period, while the organic and biochemical experiments may require up to 3 hours to finish. Suggestions are occasionally given to either delete some sections, or perform the experiment in pairs or groups. Virtually every experiment can be stopped at many points allowing for flexibility in varying laboratory times.

The format is the same for each experiment: a Materials Needed section lists all supplies and equipment on a per student basis. Particular care is taken to avoid dangerous or expensive chemicals. The Background reviews the theoretical aspects of the topic and the Procedure gives not only the instructions to carry out the experiment, but also suggests demonstrations and cautions on possible safety matters. Finally the Report Sheet provides an easy means both to collect the data and to guide the interpretation process.

We wish to acknowledge the support received from the Indiana University Foundation in the form of two course development grants.

Happy experimenting!

Erwin Boschmann
Norman Wells

TO THE STUDENT

The experiments in this book have been developed to give you 'hands-on' exposure to scientific methods and to chemical principles. It is hoped that through such experience the material discussed in lecture and in your text will be emphasized in a practical way. You as a consumer of goods will also become more aware of the properties of some of the multitude of products available in the modern world, and be able to make intelligent decisions regarding the use of these materials.

You should prepare yourself for the experiment before entering the laboratory, for only by doing so will you be able to make efficient use of the time. It will usually be sufficient to carefully read the experiment, but at times you may benefit from some outside reading to more fully understand the principles involved. In doing the experiments, you will learn to follow directions explicitly—a task you will be called upon many times in your professional life. This will be of value not only in chemistry, but in other areas as well. Once in the laboratory, work within the limits of the safety rules!

The Report Sheets at the end of the experiments contain a place for recording the data you take in the laboratory. This data should be recorded *directly* onto the Report Sheet, in INK, at the time you take the data. The rest of the Report Sheet may be prepared later, and you may wish to perform the calculations first on scratch paper before transferring them in ink onto the Report Sheet. It is important that you use *your* data to carry out all calculations, even if the results are not what you think they should be. You will be graded on how you handle the data, and not so much on the actual result. You must therefore *show all calculation set-ups* (not the actual arithmetic). The Report Sheets are to be removed from the book and handed in at the next laboratory period, or other specified time.

Erwin Boschmann
Norman Wells

SAFETY IN THE LABORATORY*

These experiments have been written with the necessary safety precautions in mind. Any hazards that may arise are due to those persons who offend the common rules of safety. Your safety and the safety of those about you is *your* personal responsibility. *Be safety conscious.*

General Rules

1. You must protect your eyes with safety glasses at all times and the use of a laboratory apron is recommended. Long hair must be confined to the back of the head with some type of fastener.
2. Understand your experiments and follow the procedures carefully. No unassigned experiments will be tolerated.
3. All bottles containing chemicals should be plainly labelled. Materials found in unlabelled bottles should be turned over to the laboratory instructor.
4. Unless a reagent is definitely known to be non-toxic and non-corrosive, handle it as though it were toxic and corrosive.
5. Read carefully the labels on reagent bottles. Be certain you are using the specified quantity of the right reagent in the correct concentration. A wrong choice may result in serious consequences.
6. Never use your mouth in pipetting poisonous liquids. Use a pipet bulb. Consult the assistant if uncertain about the nature of a liquid.
7. Drinking from beakers is extremely dangerous. Serious, even fatal, poisoning may result from insufficient washing of a beaker used as a drinking glass or from inadvertently drinking from a beaker containing a poisonous solution. All eating or drinking must be done outside the laboratory and should be preceded by a thorough washing of hands.
8. Smoking is not permitted in laboratories or storerooms.
9. Accidents caused by careless handling of glassware can be avoided by observing the following precautions:
   a) Fire-polish the ends of all glass rods and tubing.
   b) Before inserting glass tubing or thermometers into stoppers (or rubber tubing), be sure that the hole is large enough to accommodate the glass. Moisten both the glass and stopper thoroughly. Hold the tubing between the thumb and forefinger, *not in the palm of the hand.* Grasp the glass close to the end that is being inserted and twist the tube with even pressure. Glycerine is used as a lubricant with cork or if anhydrous conditions are necessary.
   c) Cut rubber or cork away from glass to which they have hardened. Do not push or pull them.

*Written in conjunction with P. A. Boaz, Department of Chemistry, Indiana University-Purdue University at Indianapolis.

d) When picking up a beaker, place your fingers around the outside of the bottom, not over the top rim. Small beakers of hot solution are best picked up by means of a folded paper strip.
e) Beware of hot glass. Glass cools slowly and may be hot enough to cause painful burns without appearing to be so.
f) Throw away cracked and chipped glassware immediately and obtain replacements from the instructor.

10. Flammable, volatile liquids such as alcohol, ether, benzene, etc. must never be distilled or evaporated over open flames. No flames must be in the immediate neighborhood when these liquids are in use. Carry out operations with flammable volatile substances in the hood. The hood is also used whenever toxic or irritating gases are likely to be evolved.
11. All solid waste materials are disposed of in the containers provided, *never in the sink*. Glass and metal are disposed of separately in the designated container.
12. Liquid wastes, unless toxic or corrosive, are poured into sinks while flushing with water. However, volatile liquids should not be disposed of in the sink because of the possibility of the formation of gas pockets which may blow back and ignite. Special containers will be provided for these wastes.
13. Know the location of the first aid station, fire extinguisher, fire blanket, safety showers, fire alarm, and nearest exit.
14. Clean up all spills at once. Most laboratory floors are slippery when wet. Take care to dry them after cleaning.
15. Plenty of running water is the best first aid treatment for all acid or alkali accidents to skin, clothing or laboratory furniture. *Rapid and immediate treatment is absolutely essential.* Use plenty of running water; a little water or a damp cloth may cause further damage because of heating effects. Eye injuries whether chemical or mechanical must always be considered serious. In case of chemical injury, the eyes must be forced open immediately and flushed thoroughly with water. *No other treatment is to be given except by a doctor.* Clothing soaked with strong acid or alkali should be removed. The following apply to clothing, laboratory furniture and all parts of the body *except mouth and eyes:*

    Acids—after acid has been washed off with plenty of water, solid sodium bicarbonate (baking soda) is applied liberally to the area.

    Alkalies—After alkali has been washed off with plenty of water, dilute acetic acid is applied to the area.
16. The shower is intended for use in case corrosive chemicals are spilled or splashed over a large body area. Do not use the shower to extinguish clothing on fire.
17. The most painful and serious laboratory accidents are caused by clothing catching fire. Such accidents may produce fatal results in a few seconds since the chances of recovery diminish with the extent of skin surface burned. If a person's clothing catches fire, throw him to the floor and roll him to smother the flames quickly. Do not allow him to remain in a standing position even if you must trip or knock him down. This action will prevent injury to respiratory passages and eyes by flames which naturally rise and envelop the head. *Never turn a fire extinguisher of any type on a person whose clothing is on fire.* Eye injury may result from a soda acid type and frost bite from the carbon dioxide type.
18. Small laboratory fires are usually best smothered with a small cloth, larger fires with the carbon dioxide fire extinguisher.
19. *Report all accidents to your instructor.* Give full details concerning the nature of the accident, the chemicals you were using and the immediate first aid you have taken. Only the instructor administers treatment involving materials in the first aid station.

Laboratory Manners

1. Never take stock bottles to your desk.
2. Insert nothing into bottles of solid reagent bottles. Pour the estimated amount on a piece of weighing paper or into a beaker. Discard any excess. Do not return to the stock bottle.

3. Pipets are not inserted into bottles of liquid reagents. Pour the estimated quantity into a beaker or graduated cylinder. Discard any excess. Do not return to the stock bottle. Wipe away drips from the neck and sides of these bottles.
4. Never weigh chemicals directly on the balance pan. Use weighing paper or a watch glass.
5. Distilled water is expensive and must not be wasted. It is used only for making up solutions and an occasional procedure called for by the experiment or the instructor.
6. Before leaving the laboratory, make sure your working surface is clean and dry. Be sure the water, gas, and air cocks are completely shut off.

SAFETY PLEDGE

1. Wear approved eye protection in the laboratory continuously. (If you should get a chemical in your eye, wash with flowing water from sink for 15–20 minutes.)
2. Perform no unauthorized experiments.
3. In case of fire or accident, call the instructor at once. (Note location of fire extinguisher and safety shower now so that you can use it if needed. Wet towels are very efficient for smothering fires.)
4. You must call the instructor for treatment of cuts, burns, or inhalation of fumes.
5. Do not taste anything in the laboratory. (This applies to food as well as chemicals. Do not use the laboratory as an eating place and do not eat or drink from laboratory glassware).
6. Exercise great care in noting the odor of fumes and avoid breathing fumes of any kind.
7. Do not use mouth suction for filling pipets with chemical reagents. Use a pipet bulb.
8. Don't force glass tubing into rubber stoppers. Use lubricant and protect your hands with a towel when inserting tubing into stoppers.
9. Confine long hair when in the laboratory. (Also, a laboratory apron is essential when you are wearing easily combustible clothing. Such an apron affords desirable protection on all occasions.)
10. Never work in the laboratory alone.
11. No smoking in the laboratory.

I have read the above rules and will observe them in my chemistry course.

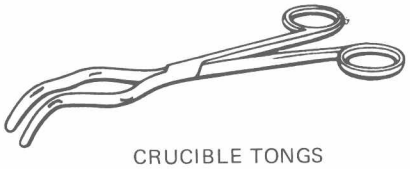
_____

signature

_____

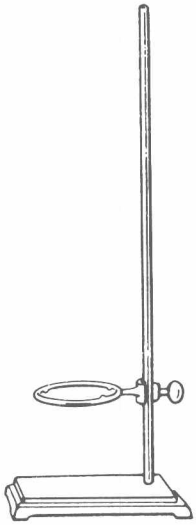
date

COMMON LABORATORY EQUIPMENT

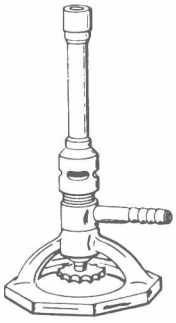


CRUCIBLE TONGS

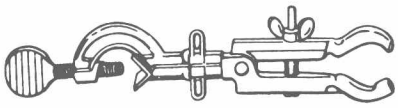
RING SUPPORT



RING STAND

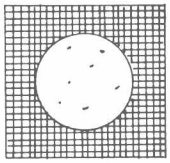


BUNSEN BURNER
(TIRRILL TYPE)

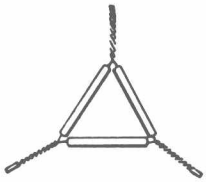


UTILITY CLAMP

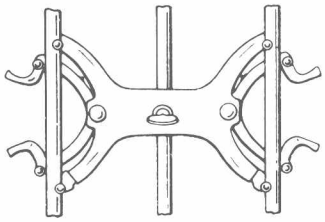
SPATULA



WIRE GAUZE



CLAY TRIANGLE

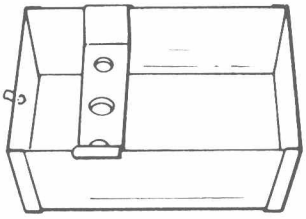


BURET CLAMP

WING TOP, FISHTAIL,
OR FLAME SPREADER

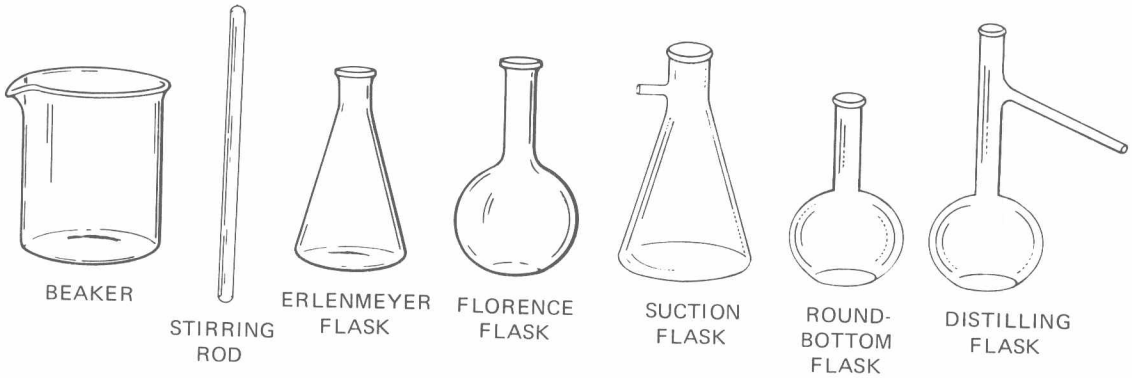
TEST TUBE HOLDER

EVAPORATING DISH



PNEUMATIC TROUGH

WATCH GLASS


BEAKER

STIRRING ROD

ERLENMEYER FLASK

FLORENCE FLASK

SUCTION FLASK

ROUND-BOTTOM FLASK

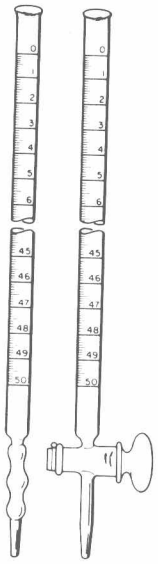
DISTILLING FLASK


THERMOMETER

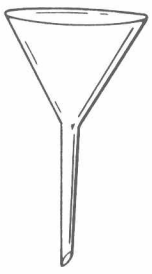

WASH BOTTLE


WIDE-MOUTH BOTTLE


TEST TUBES


BURETS


PIPET


FUNNEL

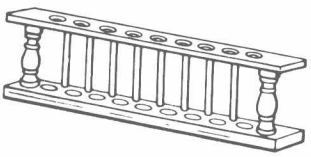

BUCHNER FUNNEL


DEFLAGRATION SPOON


GRADUATED CYLINDER

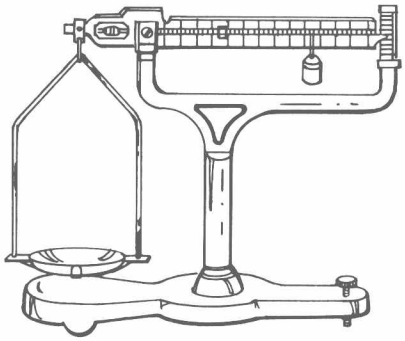

TRIANGULAR FILE


TEST TUBE BRUSH


TEST TUBE RACK


CRUCIBLE AND COVER

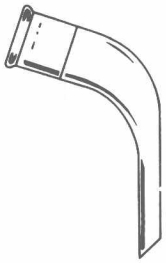

MEDICINE EYEDROPPER



TRIPLE-BEAM BALANCE

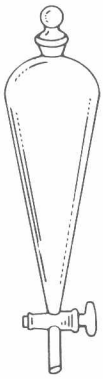
FORCEPS

MORTAR
AND PESTLE

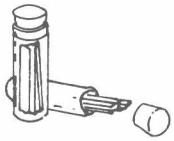


ADAPTER

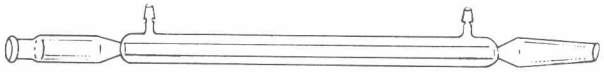
CORK BORERS



SEPARATORY FUNNEL

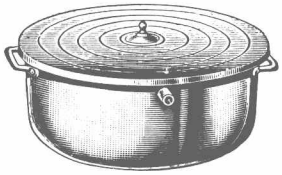


LITMUS PAPER,
RED AND BLUE



CONDENSER

GLASS PLATE



STEAM BATH

SCOOPULA

CONTENTS

INTRODUCTION TO THE LABORATORY

Materials Needed

Bunsen burner
Glass tubing
File
Wire gauze

Background

The laboratory assigned to you is equipped with tools and safety devices necessary to carry out all experiments in an efficient and safe manner. Most of these will be new to you; as you look around you might at first feel as though you are being asked to bake a cake in a totally strange kitchen where you don't know the location of any ingredient. It is the purpose of this Introduction to familiarize you with some of the physical facilities, equipment, and safety devices associated with the laboratory. While you will not be asked to hand in a report, it is important that all parts be carried out carefully and conscientiously.

Procedure

I. CHECK-IN

As soon as you have been assigned a working area and an equipment drawer, you should familiarize yourself with the contents of the drawer. To do so, transfer all items onto the desk top and identify each by name, using the drawings in the manual if necessary. Examine each item carefully for cleanliness and damage. Then check the items against the list of equipment that will be provided, making a note of any missing or damaged items. Return all usable equipment to the drawer.

II. SURROUNDINGS

1. *Reagent and Equipment Shelves.* Familiarize yourself with all storage places for equipment, reagents, and solutions in the laboratory. These items will normally be found in drawers or on shelves or benches located to the sides or at the ends of the laboratory.

2. *Stockroom.* A supply room is usually located in the vicinity of the laboratory. Become familiar with its location, operation, and staff. If the examination of your equipment showed

some damaged or missing items, you may now want to exchange these items and complete your list through the storeroom.

3. *Other.* You should also become knowledgeable with the operation of service outlets on the workbench. These usually include tap water, steam or hot water, gas, forced air, aspirator vacuum line, and a 110 V line. Distilled water may be built in or supplied separately.

Naturally, there are many other features in the laboratory. These will become familiar to you as the term progresses. There is, however, one feature that must be dealt with from the beginning.

III. SAFETY

Because you will often experiment with matter undergoing a change, safety precautions must be strictly observed to keep such changes under control. Read and then sign the Safety Pledge, which will commit you to observe the rules.

Here are the basic DOs and DON'Ts:

DO wear safety (or prescription) glasses
wear protective clothing
be clean in all work
double check the labeling on containers
see your instructor in case of any accident
put broken glass in the special container only

DON'T perform unauthorized experiments
taste anything
force glass tubing into rubber stoppers
work in the laboratory alone
eat, smoke, or drink in the laboratory
put solid wastes in the sink
take reagent bottles to your work area

Familiarize yourself with the location and operation of fire extinguishers, safety showers, fire alarm, fire safety blankets, exhaust hoods, first aid station, exits, eye-wash solutions, sodium bicarbonate for acid and base burns, the nearest telephone, and the school's emergency procedures in general.

IV. SOME BASIC TECHNIQUES

1. *The Laboratory Burner.* Many experiments require a source of heat, which is usually supplied by a bunsen burner. The common burner mixes gas with air at the base and allows the mixture to burn at its top. The size, color, and temperature of the flame are controlled by the ratio of gas to air.

Examine all parts of the burner. Note that the amount of *gas* is controlled at the base of the burner by means of a needle valve and/or at the line outlet. Note further that the amount of *air* mixed with the gas is controlled by turning the barrel, thereby opening or closing the air vents. To examine its function, remove the barrel and light the burner at the base. Observe the flame. Now turn off the gas and replace the barrel. ***Care:*** The base may be hot. Light the burner by opening the gas lines and holding a match to the top of the burner. Strikers may be used in place of matches.

First change the forcefulness of the flame by increasing and then decreasing the supply of gas. Normally, a medium flow is most suitable. Now change the appearance (and heat) of the flame by increasing and decreasing the amount of air admitted. When just a little air is allowed to mix with the gas, the flame becomes tall, blue, and relatively cool. On the other hand, when