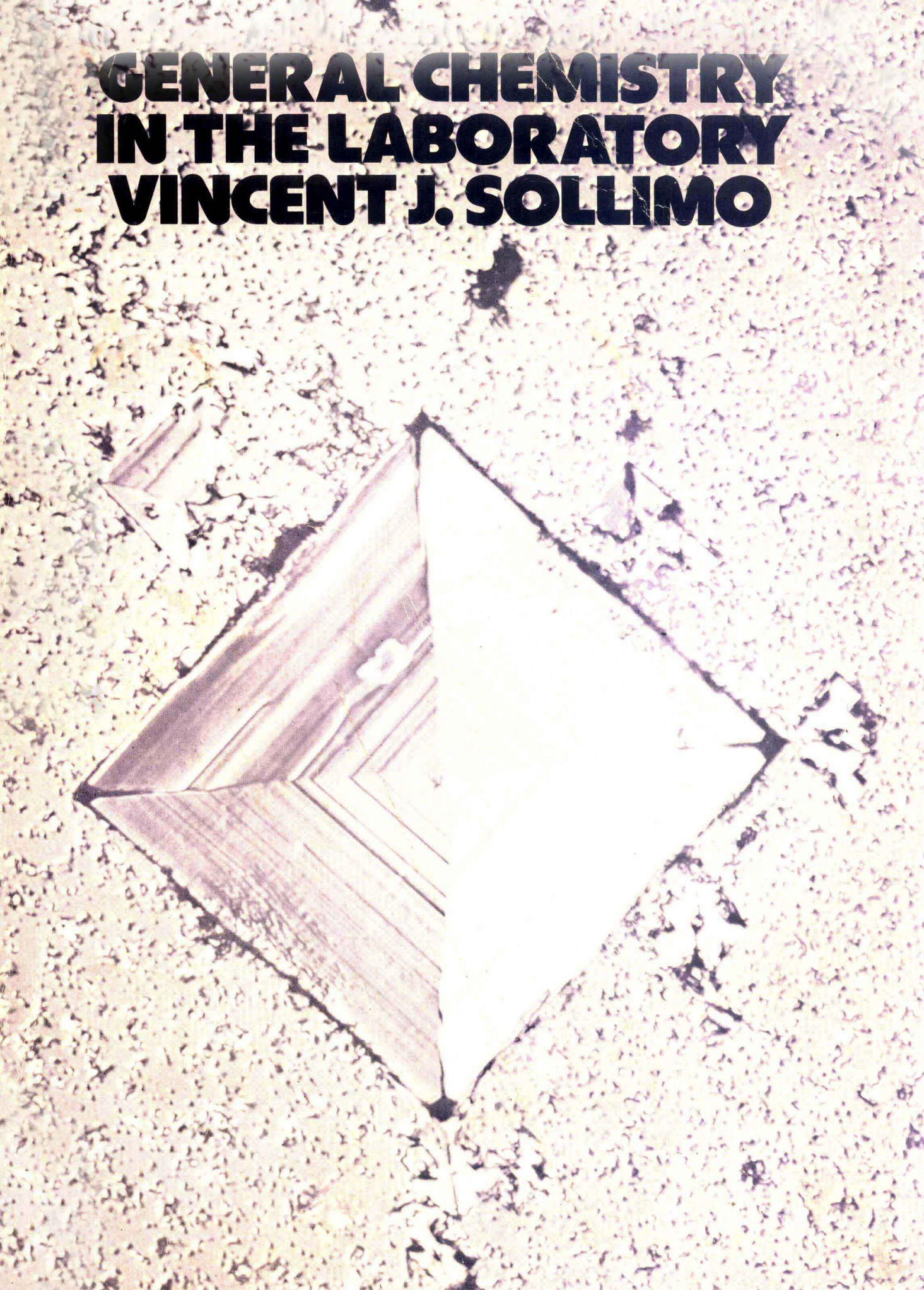
GENERAL CHEMISTRY IN THE LABORATORY

VINCENT J. SOLLIMO



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PREFACE

While College Chemistry in the Laboratory is a two-semester manual designed for students majoring in the physical and biological sciences, engineering and allied health it may also be used for the non-science major.

The experiments are listed in a widely accepted sequence but may easily be changed depending upon instructor preference. A balance of quantitative and qualitative experiments have been selected which stress the relationship between observations, data and chemical principles.

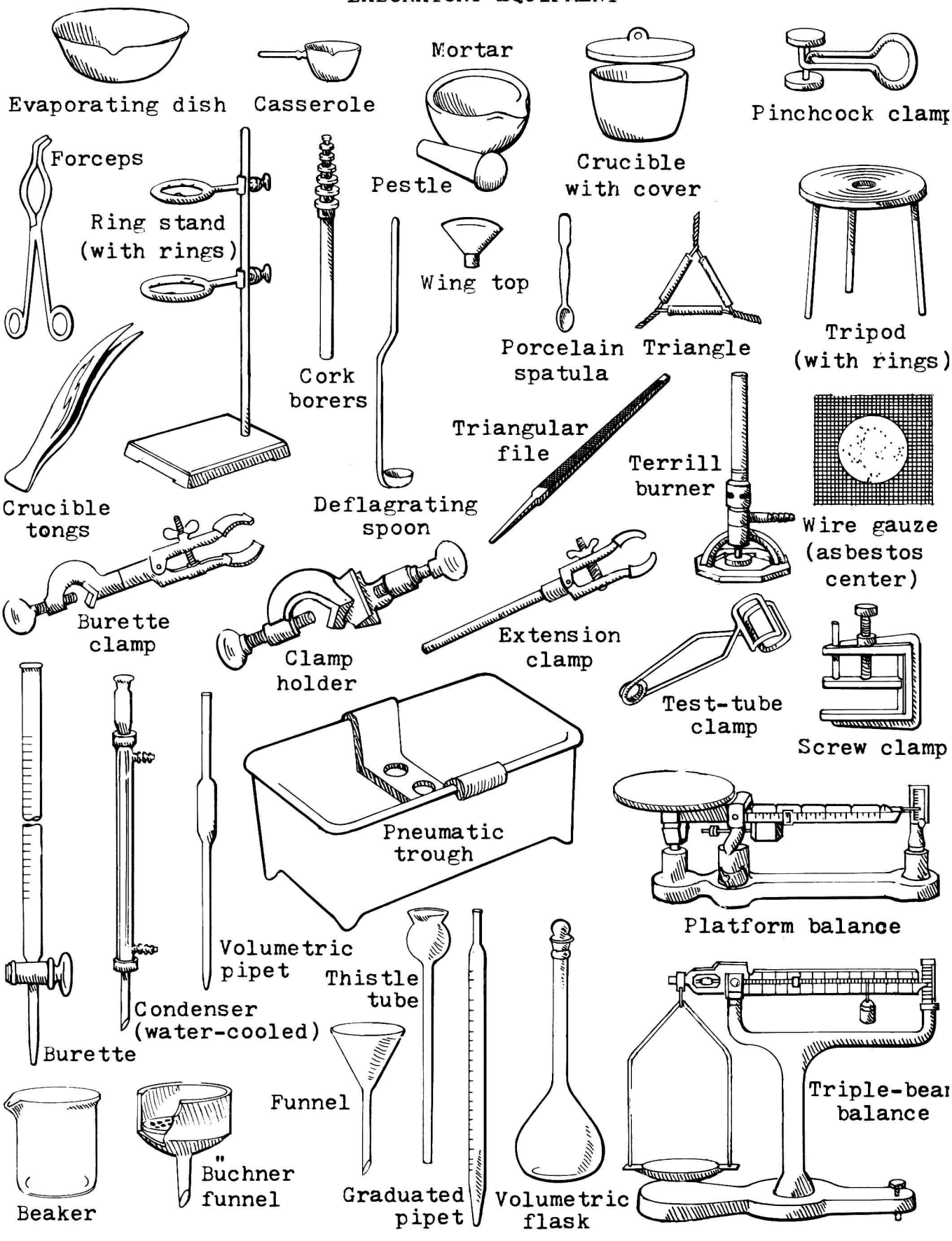
The experiments designed with safety and simplicity in mind require ordinary and readily available apparatus and materials found in general chemistry laboratories. Each experiment contains a stated goal, a list of required materials, an introduction with a theoretical background where appropriate, a procedure and a tear-out report sheet which includes data tables and questions designed to stimulate student interpretation of data. Where deemed appropriate a special effort to incorporate relevance and a reasonable reading level was made.

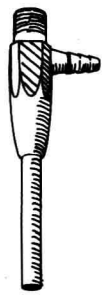
The Instructor's Guide to Experiments contains a Laboratory Safety Information Guide to Literature pertaining to toxic and hazardous materials, accident prevention guidelines and safety equipment including a list of available safety kits designed to be used for common laboratory accidents. This may be found in Appendix XIV.

The author wishes to express his appreciation to Dr. Edwin Vickner of Gloucester County College who contributed experiments 41, 42 and 43 and Glenn Malsbury for his artistic talents in graphic illustrations. A special note of appreciation is due to the author's wife, Kay, Donald C. Jackson, College Chemistry Editor of McGraw-Hill Book Company and Dr. David Adams of North Shore Community College for their professional guidance in the preparation of this manual.

Vincent J. Sollimo

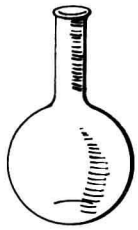
LABORATORY EQUIPMENT



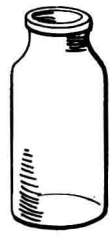


Water aspirator

Reagent bottle



Florence flask



Wide-mouthed bottle

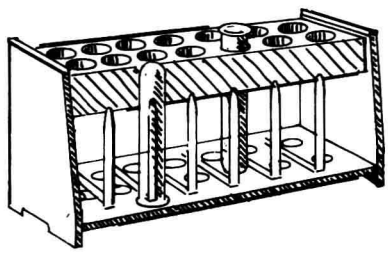
Separatory funnel

Filter flask

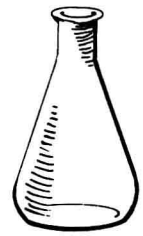
Watch glass

Straight drying tube

Graduated cylinder

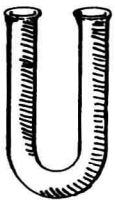


Test tubes and rack



Erlenmeyer flask

Thermometer



U-shaped drying tube

Test-tube brush

Dropping pipet (medicine dropper)

CONTENTS

Safety in the Laboratory

The laboratory is normally a safe place to work. Accidents occur because of carelessness, rushing or lack or awareness of proper techniques. Please read these safety regulations carefully.

1. Always wear safety glasses or goggles in the laboratory. Chemicals, hot liquids and glass can blind, burn or scar.

2. Find out where the fire extinguisher, safety shower, eye wash and first aid cabinet are located and how to operate these safety devices.

3. Never pipet by mouth. Use a pipet bulb.

4. Never taste a chemical.

5. Acid spills should be neutralized with sodium hydrogen carbonate and then with water.

6. Base spills should be neutralized with boric acid and then with water.

7. Smoking or eating is prohibited in the laboratory.

8. Be certain that heated glass is cooled before touching. Do not rest heated glass on table tops; use a wire gauze square with an asbestos center provided.

9. Always lubricate glass tubing when pushing it through a stopper. Cover the tubing with a towel in case the tubing breaks.

General Procedures

1. Never return any unused liquid or solid to the stock bottle. Pour the excess liquid down the drain and flush with water. Solids are disposed of in containers provided for that purpose. Never dispose of paper, glass or any solids in a sink.

2. Use the reagent bottles at their place. Never take a reagent bottle to your lab table.

EXPERIMENT 1

LABORATORY TECHNIQUES

Goal: to experience several basic laboratory techniques involving the bunsen burner, glass cutting and bending, a burette or graduated cylinder, the balance and filtering apparatus.

Apparatus and reagents:
square wire gauze,
forceps,
6 mm glass tubing,
triangular file,
wing-top burner,
one iron ring,
125 ml Erlenmeyer flask,
50 ml graduated cylinder,
50 ml burette,
burette stand and clamp,
one 50 ml beaker,
one 400 ml beaker,
funnel,
stirring rod,
filter paper,
1 M sodium sulfate, and
1 M barium nitrate

BACKGROUND:

Good laboratory technique is essential to obtaining good data and results. Be familiar with equipment and glassware as outlined in this manual. The techniques introduced in this exercise are introductory. You will learn more techniques in later experiments. Before beginning the techniques keep the following in mind:

1. When smelling vapors in a container never hold your nose close to the source. Instead, waft the vapors toward you by moving a cupped hand across the opened container.

2. Only certain pieces of glassware can be heated such as test tubes, crucibles, beakers and evaporating dishes. Never heat graduated cylinders or watchglasses and never place a thermometer in a flame. Thermometers should be placed in heated liquids, gases or solids in such a manner that the flame is not in close contact with them. When making measurements always take the most accurate reading that an instrument permits. If a balance can determine mass within ± 0.01 g, you should record that mass to within 0.01 g.

Depending upon the instrument you use, different degrees of accuracy will be obtained. Table 1-1 lists the accuracy attained with some common laboratory measuring devices.

The number of significant figures in a measurement will determine its accuracy. In adding the masses of the three samples below the answer is rounded off to the tenths place because Sample 3 is accurate only to that place. For a more detailed description of significant figures read Appendix II.

Sample 1:	2.036	g
Sample 2:	2.1	g
Sample 3:	0.1	g
	4.2	g

Table 1-1

Accuracy for Common Measuring Devices	
Instrument	Accuracy
metric ruler	± 0.1 cm
graduated cylinder	± 0.1 ml
burette	± 0.02 ml
platform balance	± 0.1 g
triple beam balance	± 0.01 g
top loading balance	± 0.01 g and ± 0.001 g
analytical balance	± 0.0001 g

PROCEDURE:

Wear safety goggles!

1. <u>The Burner</u>.

Figure 1-1 illustrates the key parts of the burner.

A Typical Burner

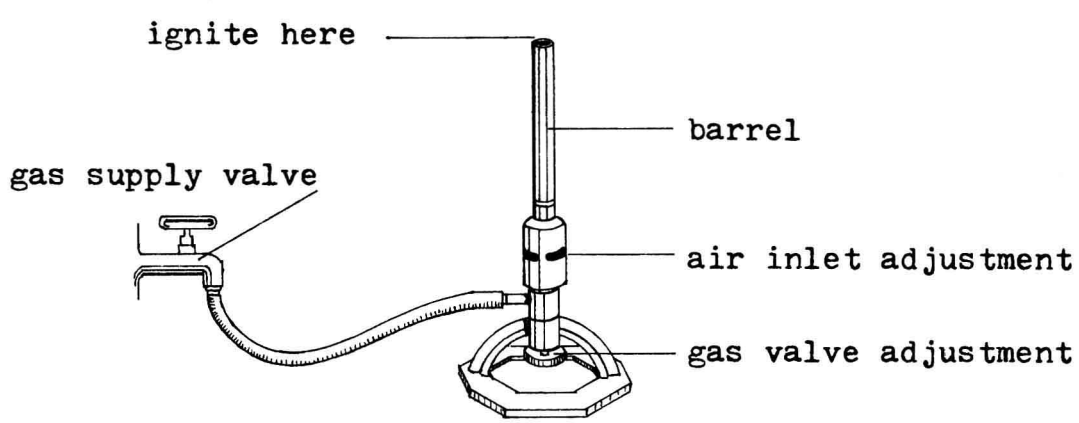


Figure 1-1

A. Close the gas adjustment valve completely.
B. Open the gas supply valve completely.
C. Open the air inlet adjustment to half way.

D. Open the gas adjustment valve slightly, and with a stricker or match ignite the air-gas mixture on the side of the gas flow and not over the mouth of the barrel.

E. Adjust the flame height with the gas adjustment valve.

F. Close the air inlet adjustment and place a square wire gauze held by a pair of forceps in the region above the inner cone as shown in Figure 1-2. Now open the air inlet adjustment as much as possible without having the flame blow out. Place the wire gauze above the inner cone again. Record your observations describing the color of the cones and relative temperatures.

Cones of a Bunsen Burner

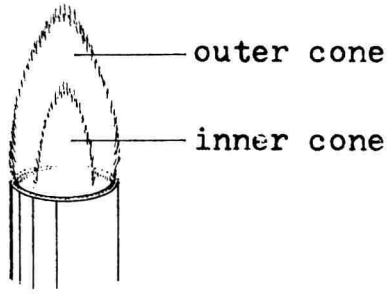


Figure 1-2

2. Glass Tubing Cutting.

Take a 40 cm length of 6 mm diameter glass tubing and using a triangular file make a scratch in the center of the tubing by rapidly moving the file forward in a single stroke. Avoid "sawing" the glass tubing with the file. Holding both thumbs opposite the scratch which faces away from you, press away with the thumbs while pulling slightly with the hands. The glass tubing should snap like a pretzel along the scratch. If not, make a deeper scratch.

3. Fire Polishing.

To prevent jagged edges from cutting you or getting stuck in a stopper fire polish both ends of the tubing. Rotate the rough edges in the flame as shown in Figure 1-3 until the glass becomes soft and the sharp edges become rounded and smooth. Remove it quickly after rounding before the hole becomes too constricted.

Rest the hot tubing on a special wire gauze which will ab-

sorb the heat and permit the hot glass to cool more quickly. Never place a hot object on a lab table! Be certain to wait until the glass cools before picking it up. A good practice is to always pick up the glass at the cool ends.

Fire Polishing

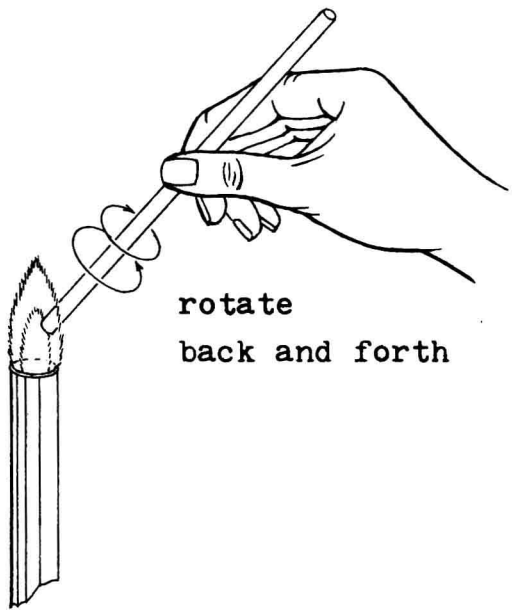


Figure 1-3

4. Glass Tubing Bending.

A. Attach a wing top to your burner. Be careful that the burner has had suffiecient time to cool. Turn on the burner and rotate a 20 cm piece of tubing, from Part 2 above, in the flame as shown in Figure 1-4. Continue to rotate the tubing until the glass becomes soft and begins to sag.

Heating Glass Tubing

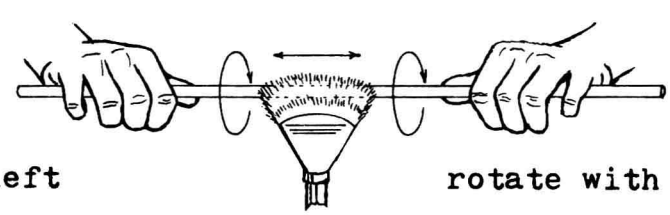
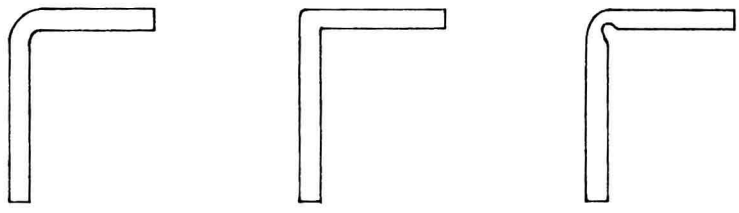


Figure 1-4

B. When the glass becomes soft and sags slightly in the center remove it from the flame and slowly bend it into a 90° angle as shown in Figure 1-5. Repeat with your second piece of tubing.

A 90° Bend



a good bend a poor bend a poor bend

Figure 1-5

5. Insertion of Tubing into a Rubber Stopper.

Ask your instructor for a one-hole rubber stopper that will permit the insertion of the fire-polished glass prepared above. Wet the stopper hole and the tubing with water or glycerol as a lubricant. Using towels to hold the stopper in one hand and the tubing in the other, slowly and gently twist the tubing through the hole until about 3 cm comes through the stopper. Be certain the toweling covers both hands to protect them from cuts in the event the glass tubing breaks.

6. Measuring Length.

Measure the length of a small test tube in centimeters. Record this in your data table to the closest tenth of a centimeter.

7. Measuring Volume.

A. Because water is attracted to glass a meniscus is formed along the sides of a water container as shown in Figure 1-6. All volumes should be read at the bottom of the meniscus when the meniscus is at the same height as the eye of the observer.

B. Fill a 125 ml Erlenmeyer flask with water and using a graduated cylinder measure its volume to the nearest 0.1 ml. Repeat using a small test tube. Record this in your data table.

C. Fill a burette until the meniscus is at the 0.00 mark. Practice controlling the rate of flow of water from the