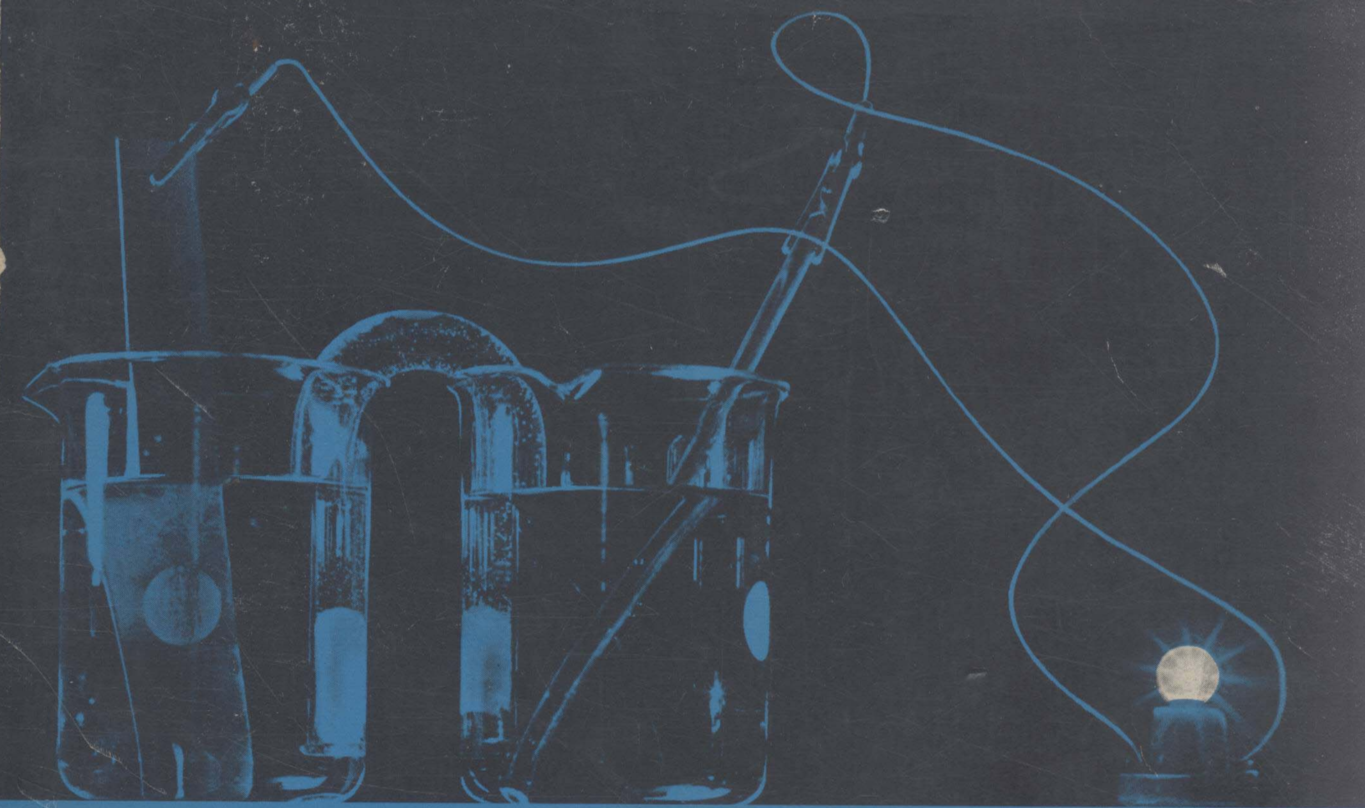

CHEMISTRY

AN EXPERIMENTAL SCIENCE

Laboratory Manual

CHEMICAL EDUCATION MATERIAL STUDY

Laboratory Manual for

CHEMISTRY

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CHEMICAL EDUCATION MATERIAL STUDY

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LABORATORY MANUAL FOR

CHEMISTRY

AN EXPERIMENTAL SCIENCE

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An Experimental Science

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PREFACE

The CHEM Study course approaches the study of chemistry as an experimental science. It is a *laboratory-centered* course which:

(1) features experiments which will permit you to make your own discoveries of the regularities and principles which unify chemistry and make it easier to understand,

(2) emphasizes the making of careful observations and quantitative measurements under controlled experimental conditions,

(3) stresses the preparation of well-organized tables for recording data and the results of calculations so that you can more readily make deductions and recognize the regularities which exist,

(4) uses challenging discussion questions which will help you to apply the principles observed in the experiments to new situations.

The Textbook discussions are closely meshed with the laboratory work. Each chapter in the Textbook is preceded by one or more experiments which provide an experimental background for the topics discussed. As you learn to recognize and use the important principles you will be in a better position to appreciate and understand the theories which have been proposed to explain the regularities in chemistry.

The organization of the CHEM Study course is revealed by the five major divisions into which the experiments have been classified:

Part I. Observation and Interpretation. Precision of Measurement. (Six experiments)

Part II. An Introduction to Chemistry. The Mole Concept. Avogadro's Hypothesis. Gases. Solutions. (Eight experiments)

Part III. Investigations of Chemical Reactions Illustrating Important Principles. (Thirteen experiments)

Part IV. Theoretical Concepts. Atomic Structure. Chemical Bonds. Structure and Properties. (Four experiments)

Part V. Application of Chemical Principles to Descriptive Chemistry. (Fifteen experiments)

Familiarize yourself with each experiment you are to perform before coming to the laboratory. Prepare tables for recording data and calculations in a blank laboratory notebook. Read carefully the directions on page ix for writing laboratory reports. The introductory section of the manual contains two full page illustrations of laboratory apparatus you will use. Read carefully the laboratory instructions and safety precautions on page ix.

Gain self-reliance by working alone on an experiment unless directed otherwise. Use your ingenuity and common sense. You will find that there is always opportunity for logical and imaginative thinking.

Illustrated sections on lighting and adjusting a laboratory burner and manipulating glass tubing will be found in Appendixes 1 and 2. Appendixes 3, 4, and 5 deal with the metric system, experimental errors, and a brief review of some mathematics which will be useful to you in making calculations. Appendix 6 contains additional experiments which you may perform at the option of your teacher. Many experiments contain suggestions for additional investigations which may be undertaken with the permission of your teacher.

Remember that your laboratory work is the core of your chemistry course. You have a unique and challenging opportunity to observe first-hand many of the facts and regularities of chemistry. Out of such observations scientists developed the principles that unify chemistry and the theories which provide the current explanations. This is the scientific method in action.

The teachers and students who used the three trial editions of this manual have made important contributions to this edition. Because of their valuable suggestions and their participation in many evaluation sessions, we were provided with a reliable basis for the revision presented here. All members of the CHEM Study staff have contributed to the development of the experiments. In particular we wish to acknowledge the significant contributions made by the following high school teachers on our staff: Mr. Joseph E. Davis, Miramonte High School, Orinda, California, Mr. Keith MacNab, Sir Francis Drake High School, San Anselmo, California, and Miss Margaret Nicholson, Acalanes High School, Lafayette, California.

There are numberless ways in which the CHEM Study is indebted to the institutions listed below for contributions of personnel, facilities, and encouragement. Finally, we acknowledge with thanks the stimulation and support received from the National Science Foundation that made the CHEM Study possible.

January 1963

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LABORATORY INSTRUCTIONS

1. Remember at all times that the laboratory is a place for serious work.
2. Always prepare for an experiment by reading the directions in the manual before you come to the lab. Follow the directions implicitly and intelligently, noting carefully all precautions. Check any deviations contemplated with your teacher.
3. **Do only the experiments assigned or approved by your teacher. Unauthorized experiments are prohibited.**
4. If an acid or other corrosive chemical is spilled, wash it off immediately with water.
5. Do not touch chemicals with your hands unless directed to do so.
6. Never taste a chemical or solution unless directed to do so.
7. When observing the odor of a substance, do not hold your face directly over the container. Fan a little of the vapor toward you by sweeping your hand over the top of the container.
8. Allow ample time for hot glass to cool. Remember hot glass *looks* like cool glass.
9. Smother any fires with a towel. Also, be sure you know the location of the fire extinguisher in the laboratory.
10. **Report any accident, even a minor injury, to your teacher.**
11. Wear protective eye goggles when handling dangerous chemicals. Use the fume hood when directed.
12. Throw all solids and paper to be discarded into a waste jar or waste basket. Never discard matches, filter paper, or any slightly soluble solids, in the sink.
13. Check the label on a reagent bottle carefully before removing any of its contents. Read the label twice to be sure you have the right bottle.
14. Never return unused chemicals to the stock bottles. Do not put any object into a reagent bottle except the dropper with which it may be equipped.
15. Keep your apparatus and desk top clean. Avoid spillage, but if you do spill something, clean it up immediately. Put your own equipment into your drawer and return any special apparatus to its proper place at the end of the period.

LABORATORY REPORTS

1. Record all data in a laboratory notebook as soon as possible after making the observations. Make no erasures; instead, cross out any errors with a single line. Always record your name, the date, and the title of the experiment.
2. Enter all data and observations neatly. Use tabular form whenever it is appropriate. When possible, design a data table before coming to the laboratory.
3. Indicate the operations used in making calculations by showing an orderly sample calculation. Do not clutter the calculations section with arithmetic details. Indicate the units used for all measurements. Normally calculations should not be made during the laboratory period.
4. *Answer the numbered questions* wherever they appear as part of your laboratory report. Use concise statements.
5. *Do not include written answers* to questions which appear in the introduction and procedure section. Some are intended to direct your attention to problems which will be investigated. Others point out the reason for certain procedures or controls which will enable you to proceed with greater understanding.

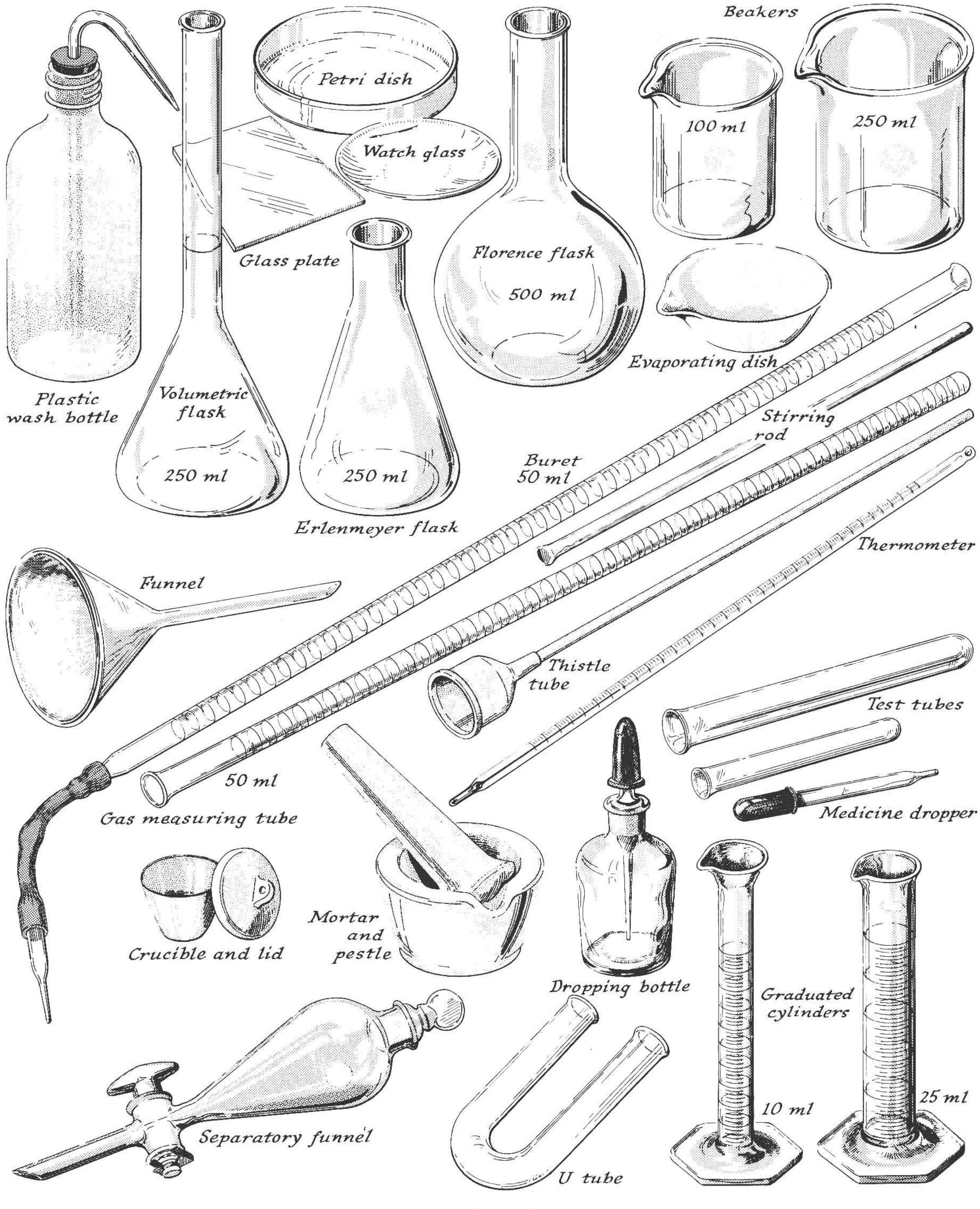

Fig. i-1. **Typical chemical laboratory apparatus**—*glass and porcelain.*

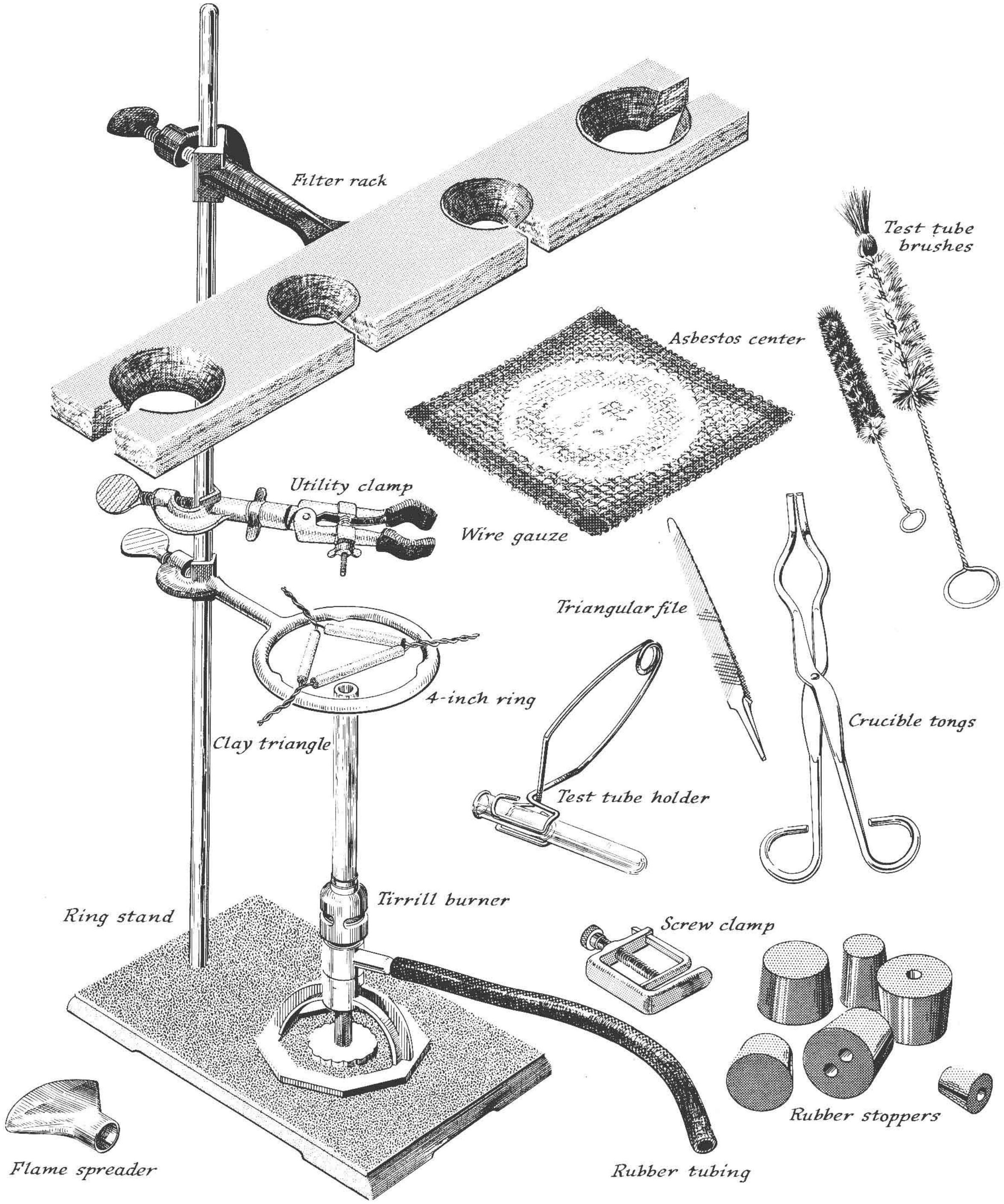

Fig. i-2. **Typical chemical laboratory apparatus** — *metal equipment and accessories.*

CONTENTS

Part IV. Theoretical Concepts. Atomic Structure. Chemical Bonds. Structure and Properties

Part V. Application of Chemical Principles to Descriptive Chemistry

I

SCIENTIFIC OBSERVATION AND DESCRIPTION

Everyone thinks of himself as a good observer. Yet there is much more to observation than meets the eye. It takes concentration, alertness to detail, ingenuity, and often just plain patience. It even takes practice! Try it yourself. See how complete a description you can write about a familiar object—say, a burning candle. Be "scientific" about this and start with an experiment. This means you should observe a burning candle in a laboratory, that is, a place where conditions can be controlled. But how do we know what conditions need be controlled? Be ready for surprises here! Sometimes the important conditions are difficult to discover but an experiment can be meaningless unless the *conditions that matter* are controlled. Here are some conditions that may be important in *some* experiments but are not important here.

The experiment is done on the second floor.
The experiment is done in the daytime.
The room lights are on.

Here are some conditions that *might* be important in your experiment.

The lab bench is near the door.
The windows are open.
You are standing close enough to the candle to breathe on it.

Why are these conditions important? Do they have something in common? Yes, there is the common factor that a candle does not operate well in a draft. Important conditions are often not as easily recognized as these. A good experimentalist pays much attention to the discovery and control of conditions that are important.

PROCEDURE

First examine the candle carefully. Then light it, and record in your notebook as many observations as you can during a short period (10–15 minutes).

Turn in the carbon copy of your observations to your teacher before you leave the laboratory.

Write a description of a burning candle, based upon your list of observations and upon any additional observations of burning candles you care to make later at home.

2

BEHAVIOR OF SOLIDS ON WARMING

Careful observation of a familiar object usually reveals characteristics not recognized before. You noted this as you studied the candle in Experiment 1. These details of observation raise questions. Let us give attention to one such question:

"What is the colorless liquid in the bowl at the top of the burning candle?"

This is a question with a ready answer. Perhaps the liquid is just melted wax. But how do you know this? What evidence can you offer? What kind of experiment would help you to decide if this ready answer is correct?

Let us proceed by comparing the behavior of several different substances when heated.

PROCEDURE

Part I

a. Place a tin can lid, tinned side up, on an iron ring attached to a ring stand as shown in Fig. 2-1. The lid should have three depressions to contain some of the substances tested—see (b). Adjust the height of the ring until the lid is about 8 centimeters (cm) above the candle (1 cm is almost $\frac{1}{2}$ inch).

b. Place on the lid, equally spaced near the edge, small, approximately equal amounts of each of the following substances (a quantity about the size of a match head is appropriate):

candle wax	lead
steel wool	tin
sulfur	copper wire
silver chloride	

Place the candle wax, sulfur, and silver chloride each in a separate depression in the lid, and space the other substances as shown in Fig. 2-1. The amount and location on the lid are *conditions that matter*.

c. Light the candle, and adjust the ring height until the tip of the flame is about 4 cm directly below the center of the lid. Heat the lid for about 3 minutes. Record your observations as you make them, paying particular attention to the melting behavior.

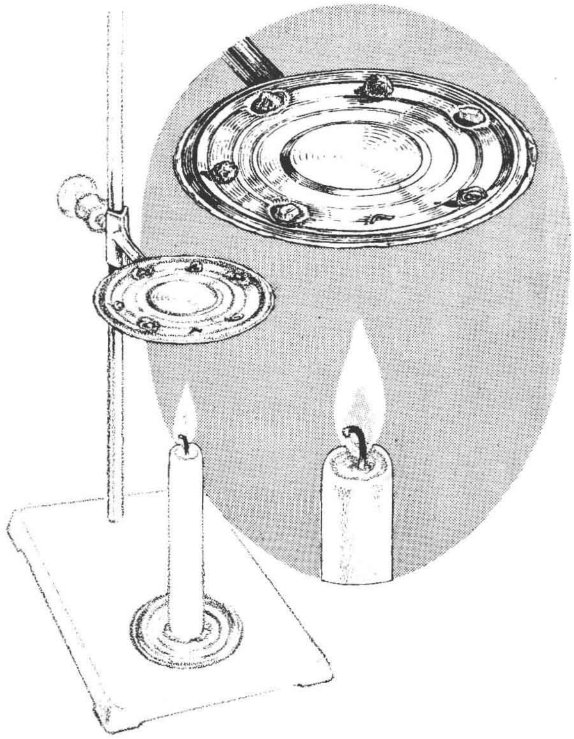

Fig. 2-1. **Arrangement of substances on a lid.**

d. Remove the candle and adjust the height of the iron ring so the lid is about 8 cm above the top of a burner. (See Appendix 1 for directions for lighting and adjusting the burner.) Heat the lid with the burner flame adjusted about 5 cm high for about 3 minutes. Increase the size of the flame and heat for several minutes more. Record your observations.

Part II

Now let us heat some of the solidified liquid from the bowl of the candle and some of the candle wax to see how they compare.

a. Remove the lid from the ring stand and replace it with a wire gauze and a 250 milliliter (ml) beaker or small tin can about one-third full of water. See Fig. 2-2.
b. Pour a few drops of the liquid from the bowl of a burning candle onto a piece of paper. Break off a small piece of the solid and place it on the water contained in the beaker or can. Obtain a piece of candle wax by cutting a chip from the bottom of your candle. Both pieces should be about the same size. Place the second piece on the water in the beaker or can apart from the first piece.
c. Heat the beaker or can and its contents with a burner flame and note when each substance starts to melt. Allow the wax to solidify, then discard it in the waste jar. *Do not pour liquid wax into the sink.*

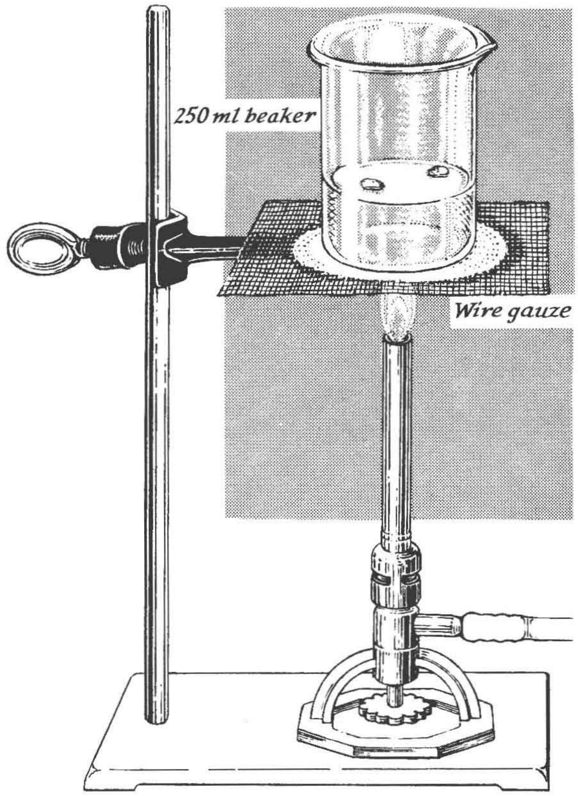

Fig. 2-2. **Comparing melting behavior.**

QUESTIONS AND EXERCISES

1. How does your observed order of melting for the substances tested compare with that observed by other members of the class?
2. Make a generalization based on the combined observations.
3. What statement can you make concerning the material in the bowl of the burning candle and the candle wax, based upon your generalization from Exercise 2?

A Question to Wonder About

Why did the substances tested on the lid begin to melt at different temperatures?

3

THE MELTING TEMPERATURE OF A PURE SUBSTANCE

The method used in Experiment 2 to determine the order in which materials melt as the temperature is raised is a satisfactory experimental technique but not a convenient one. Chemical stockrooms contain several hundred different chemicals and if we were to follow this method, we would have to place each on the lid. A more practical method is to measure separately the temperature of melting (the melting point) of each of the substances. Then these characteristic temperatures can be filed for comparison with the measured melting points of any other sample at any later time.

Until now we have been dealing only with the relative order of melting points. Now let us look at the melting of one substance more closely. A solid, *para*dichlorobenzene, will be heated slightly above its melting temperature. Temperature data will be obtained by noting the temperature at designated time intervals as the liquid cools and solidifies. The solid will then be reheated and melted, and again temperature data will be obtained at designated time intervals during the heating.

PROCEDURE

Part I. Cooling Behavior

a. To facilitate the recording of data in this experiment, you should work with a partner. One partner should prepare a table in his notebook which will allow him to record systematically the temperature, the time, and the cooling behavior. Two additional copies of the data should be made—one will be handed in; one is for the other partner (that is, there should be two copies and one original). See the sample data table at the end of this experiment.
b. Fill a 400 ml beaker three-fourths full of tap water at about room temperature (below 30°C). Place the beaker on the base of your ring stand.
c. Slowly raise the temperature of about 15 grams (g) of the moth repellant *para*dichlorobenzene in a test tube until it melts. Heat the test tube with the burner adjusted to a low flame. Place a thermometer in the liquid and continue heating until the temperature is between 65°C and 70°C. Continuously move the test tube back and forth through the flame as shown in Fig. 3-1.

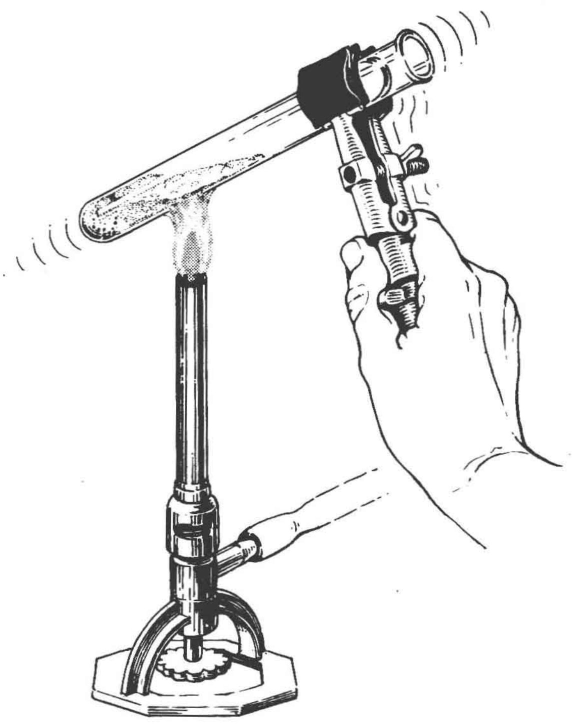

Fig. 3-1. **Melting the solid crystals.**

d. Clamp the test tube containing the melted *para*dichlorobenzene into position above the beaker of water.
e. Now clearly divide your labor. Place a clock or watch in such a way that one partner, the recorder, can read the time every 30 seconds. This partner also is to record all the observations noted by the other partner.
f. When all is ready, check the temperature of the *para*dichlorobenzene and record it to the nearest 0.2°C. On signal by the recorder, immediately immerse the lower half of the test tube into the water bath and clamp the test tube in position. Hold the thermometer against the side and just off the bottom of the test tube so that it will become imbedded in that position when the *para*dichlorobenzene solidifies (see Fig. 3-2). Record the temperature each 30 seconds until a temperature in the upper thirties is reached. Note also when solidification starts and when it is complete.

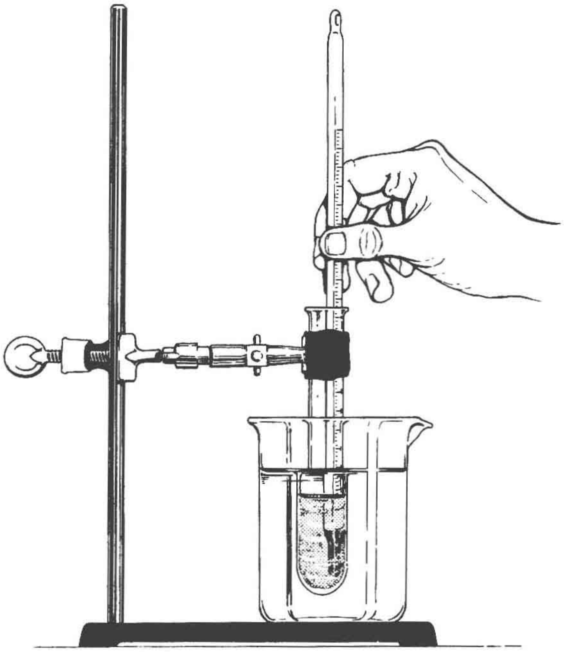

Fig. 3-2. **Apparatus for observing the cooling behavior.**

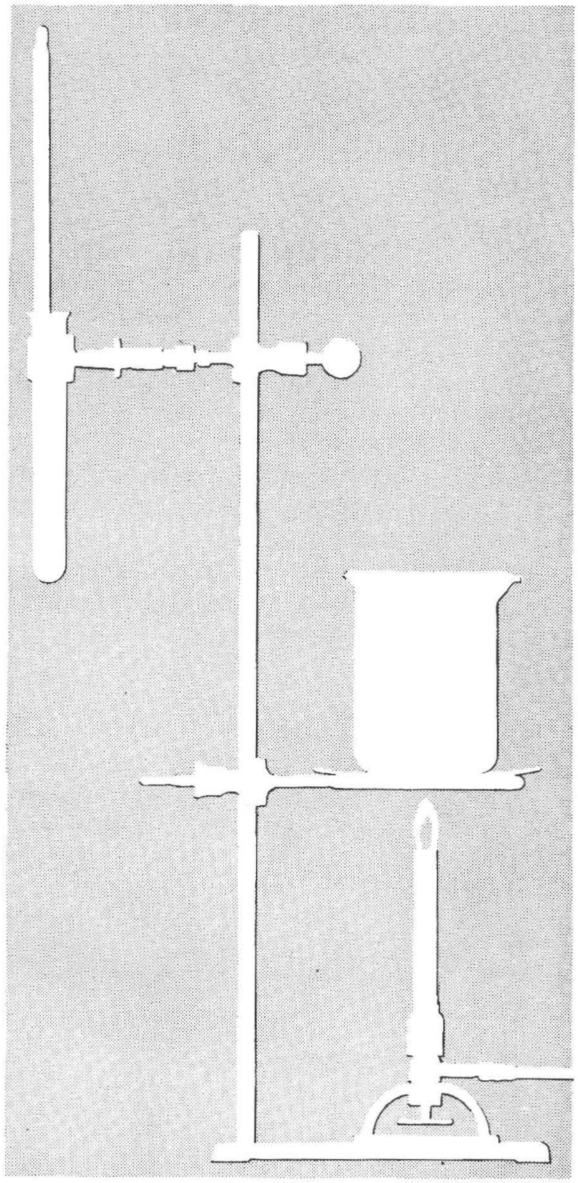

Fig. 3-3. **Apparatus for observing the warming behavior.**

Part II. Warming Behavior

The observer and recorder should exchange duties at this point.

a. Raise the test tube out of the water bath and turn the clamp around so the test tube is on the other side of the ring stand, opposite the water bath, as shown in Fig. 3-3. Place the beaker, three-fourths full of water, on an iron ring and wire gauze. Heat the water bath to approximately 70°C (start with hot tap water, if available, to save heating time), then turn off the burner, but leave it in position.
b. Record the temperature of the solidified *para*dichlorobenzene to the nearest 0.2°C. With