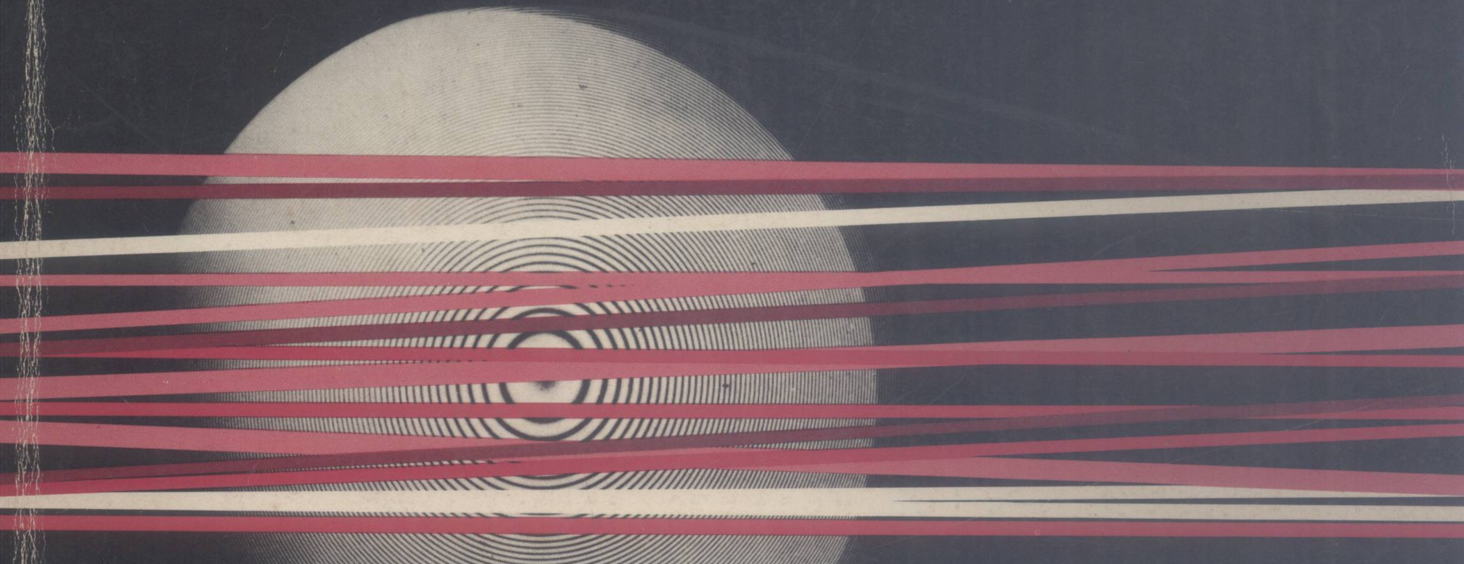
LABORATORY EXPERIMENTS FOR

INTRODUCTION TO CHEMISTRY 2ND ED.

T. R. DICKSON



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Introduction to
Chemistry
Second Edition

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Introduction to Chemistry

Preface

Chemistry is an experimental science and laboratory experience is a necessary part of the study of chemistry. Many important concepts are vitalized in laboratory exercises. Furthermore, much nonverbal learning is accomplished by the student through laboratory work. This laboratory manual is designed to be used in a first course in chemistry. It is intended for students who have had limited or no laboratory experience. Laboratory techniques, procedures, and precautions are emphasized. The collection and use of experimental data are encouraged.

This manual provides great flexibility in the laboratory experience. A variety of experiments is included so that the instructor can select the laboratory sequence that he prefers. The experiments are of a qualitative and quantitative nature. That is, some experiments stress the observations of the properties and chemical reactions of substances while others emphasize the collection of data used to carry out calculations. The student is provided with detailed directions but is also challenged by situations in which he must draw conclusions, describe observations, identify unknowns, and carry out calculations.

The first part of the manual includes safety rules and a laboratory equipment list. In Experiment 1 the student learns basic laboratory techniques and makes observations and measurements. Density is studied in Experiment 2. In Experiment 3, "Elements, Compounds and Observations," the student observes the properties of some common chemicals. Experiment 4, "Separation Methods," investigates several methods that illustrate purification and separations. Experiment 5, "The Nuts and Bolts of Chemistry," uses nuts and bolts to emphasize the meaning and use of the mole concept. Experiment 6 involves the observation of some reactions of oxygen and other elements to illustrate chemical behaviour. Experiment 7 is the quantitative determination of the empirical formula of a compound. In Experiment 8 hydrates are studied, and percentage by mass water is determined. Experiment 9 involves the preparation and observation of the properties of some common gases. This is a prelude to the study of gas laws. The "Periodic Table and Periodicity" is an exercise including the history of the table, the nature of the table, and the meaning of periodicity. In Experiment 11, "The Atmosphere," the student studies the composition of the atmosphere and determines the percentage of oxygen in air. Experiment 12 demonstrates the various gas laws. Although it is a demonstration it does provide the student with the opportunity to plot experimental data. (Appendix 2 instructs the student in graphing techniques.) Experiment 13 involves the determination of the molar mass of a volatile liquid by vapor density. Experiment 14, "Chemical Energy," studies the energy changes that accompany chemical reactions and phase changes. Experiment 15 studies solutions and solubility. "Conductivity," Experiment 16, involves electrolytes and nonelectrolytes. It is a good experiment for illustrating the nature of ionic solutions. Experiment 17 discusses some acids and bases. In "Chemical Reactions," Experiment 18, the student carries out chemical reactions and writes the net-ionic equations for the reactions. In Experiment 19 the student learns to use the buret and how to titrate an acid-base titration. A solution of sodium hydroxide is standardized and used to analyze a vinegar solution. Experiment 20 shows how, in a study of sea water, the salinity and chlorinity of a sea-water sample is determined. In "Anion Analysis," Experiment 21, the student learns the chemical tests for common anions and uses the tests to identify an unknown. In Experiment 22 the student studies the properties, models, and reactions of some organic compounds. For Experiment 23, "Radioactivity," the detection of radiation is demonstrated by the instructor, and the students perform a simulated half-life determination. The appendices include an introduction to the slide rule, graphing, and the vapor pressures of water.

I thank all the students and instructors who helped me prepare this edition of the manual. I also thank Nancy Riggs for typing the manuscript.

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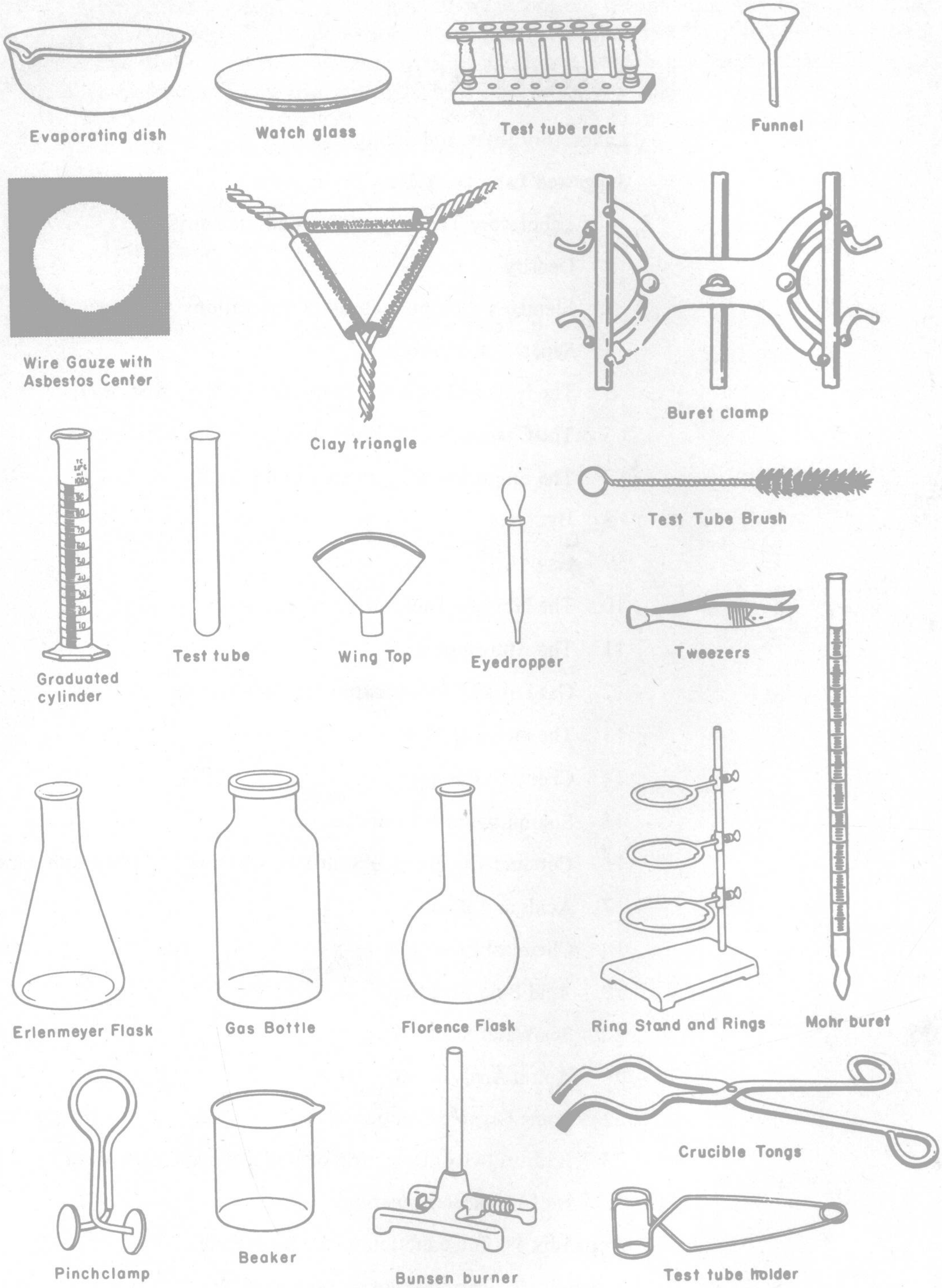
Aptos, California



Typical Laboratory Equipment

Laboratory Rules and Safety

1. Eye protection should be worn in the laboratory when you are working with chemicals or glassware.
2. Follow the instructions given in the experiments. Never perform unauthorized experiments.
3. Read all labels before using chemicals. Make sure you have the correct chemicals. Chemicals should be obtained from the reagent bottles in a clean beaker or test tube. Do not take reagent bottles to your desk. Do not take more chemicals than you need. Never return any unused material to the reagent bottle. If you take too much, leave the excess for another student or throw it away.
4. Warm test tubes and other glassware slowly before heating strongly.
5. When heating a liquid in a test tube or carrying out a reaction in a test tube, never point the mouth of the test tube at yourself or your neighbor.
6. Never taste a chemical unless directed to do so. When directed to smell a chemical, do so by wafting (fanning) the vapors toward your face with your hand. Do not place the container directly under your nose.
7. Report any injury to the instructor at once, no matter how slight it may appear to be.
8. Never pour water into concentrated acid. Always slowly pour the acid into the water while mixing.
9. If dangerous gases are given off during an experiment, do the experiment under the hood.
10. Return all waste chemicals to the containers indicated by your instructor. If no containers are available, pour all waste liquids directly down the drain and then run water down the drain. All waste solids (except those indicated) should be placed in the waste baskets. Do not throw solids in the sink.
11. Always moisten or lubricate glass tubing when it is being connected to rubber tubing or inserted in rubber stoppers.
12. As soon as you finish your experiment, disassemble the apparatus and clean any equipment used. Return any special equipment to the designated location.
13. If you spill any solid chemicals, clean them up. If you spill any liquid chemicals, clean them up with a towel. If you spill any acids or bases on the desk or floor, sprinkle some sodium hydrogen carbonate on the spill to neutralize it, then wipe it up.
14. If you splash any acid or base in your eyes, face, or hands, flush with plenty of water. Keep in mind the nearest source of water so that you can go to it as quickly as possible. If such a splash occurs, notify the instructor who will recommend any further action.
15. No eating, drinking, or smoking in the laboratory.
16. If you feel faint, sit down and lower your head between your legs.

Suggested Laboratory Desk Equipment

2 Beakers, 100 ml
1 Beaker, 250 ml
1 Beaker, 400 ml
2 Bottles, wide mouth
1 Crucible, porcelain
1 Crucible cover
1 Graduated cylinder, 10 ml
1 Graduated cylinder, 50 ml
1 Evaporating dish
2 Erlenmeyer flasks, 125 ml
2 Erlenmeyer flasks, 250 ml
1 Florence flask, 500 ml
1 Funnel
1 Test tube rack
1 Thermometer, 110°C
1 Crucible tongs
1 Tweezers
1 Test tube holder
1 Test tube brush
5 Test tubes, 15 cm
5 Test tubes, 10 cm
1 Test tube, 7 cm
2 Watch glasses
1 Clay triangle
1 wire gauze
2 Pinch clamps

Contents

1 Laboratory Techniques and Measurements

MATERIALS

Meter sticks, graduated cylinders, glass tubing.

OBJECTIVE

The purpose of this experiment is to become familiar with some laboratory techniques and equipment and to carry out measurements.

DISCUSSION

Chemistry is an experimental science and it is often necessary to make measurements and observations on objects under investigation. Quantitative measurement is fundamental to chemistry. The metric system (International System) of measurement has been established as the system of measurement used in science. The metric system is based upon defined units for mass, length, time, and temperature. A system of decimal prefixes can be used with the basic units. The unit of mass in the metric system is the kilogram (kg) but since this is a rather large mass for laboratory work, we will work most often with gram (g) amounts of materials. The unit of length in the metric system is the meter (m). In the laboratory we often express lengths in units of centimeters (cm) or millimeters (mm). Special measuring devices designed to measure a property (mass, length, time, or temperature) have been calibrated by comparison to the basic defined metric units. Length measurements are accomplished using a calibrated ruler called a meter stick. Mass measurements are carried out using a balance with which the mass of an object is compared to calibrated masses (weights) of known mass. Temperature measurement is accomplished by use of a thermometer.

It is not possible to carry out an exact measurement. Each measuring instrument is calibrated to be read to a certain number of digits. Usually a measuring instrument should be read to as many digits as possible that sometimes requires estimating between the smallest calibrated divisions on the instrument. Such an estimation process is called interpolation and is described in Fig. 1-1. Whenever you make a measurement it is very important to always read the instrument as carefully as possible and to read the proper number of digits. Furthermore, always record a measurement as a number and a unit (e.g., 2.32 cm, 5.78 g, 25.0 ml).

The numbers associated with a measurement are called significant digits. In many of the experiments in this book you will be instructed to carry out a measurement to a certain number of significant digits. For example, you may be asked to determine the mass of an object to the nearest 0.01 g. This means you are to carry out the measurements to this precision and express the result accordingly (e.g., 12.02 g, 1.32 g, 0.54 g, or 0.05 g).

Often measurements are used in calculations. Generally, a calculated result obtained by multiplication and/or division operations should not have any more significant digits than the least precise factor. For example, suppose we wanted to convert a length measurement of 2.3 in. to units of centimeters using the conversion factor

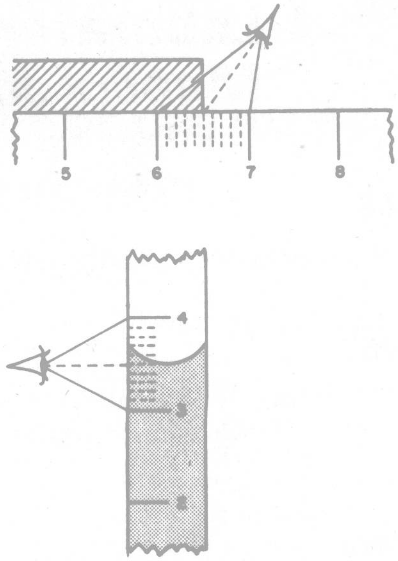


Fig. 1-1 Interpolation. The distance between divisions is visually divided into 10 equal portions and the visualized line closest to the object edge, marker, or bottom of the meniscus is noted. The distance between divisions may be divided into fewer parts depending on the calibration of the measuring device.

(2.54 cm/in.). Carrying out the multiplication to express all possible digits, 2.3 in. x 2.54 cm/in., gives 5.842 cm. Expressing the answer to this many digits is not justified since the least precise factor has only two digits. Thus, the calculated result should have only two digits: 5.8 cm. In a calculation involving multiplication and/or division, the number of digits in the result is determined by the number of significant digits in the factors and does not depend upon the position of the decimal point. When carrying out calculations in this manual, always try to express the proper number of significant digits in a calculated result.

In this manual you will sometimes be instructed to measure out a definite quantity and sometimes only an approximate quantity. It is important to note the difference. For example, you may be instructed to obtain about 2 ml of water. This means that you should obtain roughly 2 ml of water. However, if you are instructed to obtain about 2 ml of water to the nearest 0.1 ml, this means you must measure out a volume of water of about 2 ml but you must read and record the volume to the nearest 0.1 ml. The volume will not be exactly 2 ml but will be slightly larger or smaller (e.g., 1.8 ml, 2.2 ml, or 2.3 ml). Occasionally, you will be instructed to measure out a definite amount. You may be directed to measure out 2.0 ml of water. In such a case you very carefully measure out the required amount. Such a measurement can be very time consuming. Consequently, never measure out exact amounts unless you are directed to do so. However, this does not mean that you should not try to read your measuring instrument as closely as possible when you do make a precise measurement.

1. The Burner

A Bunsen burner mixes natural gas with air so that it can be burned and serve as a source of heat for laboratory experiments. The gas and air are mixed in the barrel of the burner. Note that there is an adjustable air intake vent on the barrel. The flow of gas is regulated by the gas valve connected to the gas pipe or on some burners there is a gas-regulating valve connected to the burner. To light the burner, turn on the gas valve to about half pressure (not full pressure). Hold a lighted match at the edge of the top of the barrel but not directly over the top of the barrel. If a flint striker is used, hold the striker over the top of the barrel and strike it. Once the burner has been lighted, the flame can be regulated by adjusting the gas flow and the air intake vent. The flame on a properly adjusted burner will have the appearance of a pale blue inner cone and a pale violet outer cone. The hottest part of the flame is at the tip of the inner blue cone. If there is too much gas in the flame, it will have a yellowish appearance. To obtain the proper flame, open the air intake. Too much air and gas pressure will form a flame that separates from the burner top. To adjust such a flame, lower the gas pressure and decrease the amount of air. If you want a flame that is cooler than the normal flame, decrease the amount of air by adjusting the air intake. A low burning flame can be obtained by adjusting the gas pressure using the gas valve.

Light your bunsen burner and adjust the flame to give a pale blue inner cone and a pale violet outer cone. Sketch a side view of the burner showing the flame and indicate the colors of the cones (A). Any parenthetical letter in the laboratory procedure refers to an entry on the report sheet at the end of the experiment.

2. Dispensing Chemicals

When pouring liquid chemicals from glass-stoppered reagent bottles, remove the stopper and hold it in your fingers while carefully pouring the liquid into the desired container. This technique is illustrated in Fig. 1-2. When pouring from a screw cap bottle, set the cap down on the top so that it does not become contaminated. Be sure to put the correct cap on the bottle after you have used it. If you spill any liquid or drip some on the side of the bottle, clean it up. Never pour chemicals back into the reagent bottles since they may become contaminated.

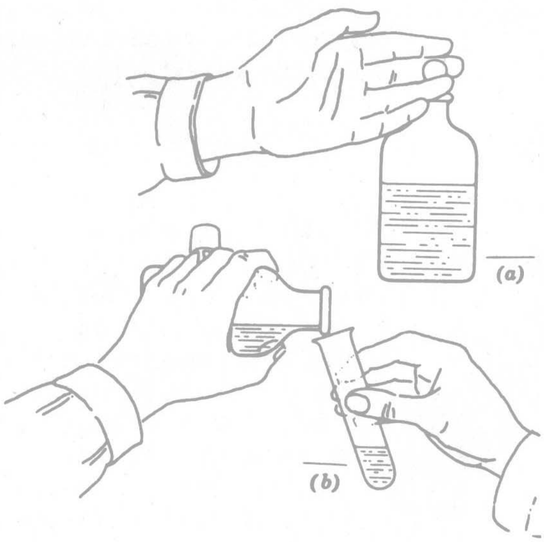


Fig. 1-2 Proper technique for pouring liquid from a glass-stoppered bottle.

When obtaining samples of powdered or crystalline solids from a jar, pour the desired amount of the solid onto a small piece of clean paper or into a clean beaker. To pour a solid do not turn the jar upside down and dump. Carefully tilt the jar and rotate it back and forth to work the solid up to the lip. Then, using the same back and forth rotation, allow the desired amount of solid to fall from the jar. This is illustrated in Fig. 1-3. Be careful when transferring a solid from a jar. If you take too much, leave the excess on a piece of paper for other students or throw the excess away. Never put any solid back into the jar. Furthermore, never put wooden splints, spatulas, or paper into a jar of solid unless your instructor indicates that this is permissible. Solids may be poured into test tubes by using a piece of paper that has been creased down the center. (See Fig. 1-3)

Take a large test tube to the designated shelf or station and pour a sample of liquid into the tube from a glass-stoppered bottle. Place the tube in your test tube rack for use in the next section.

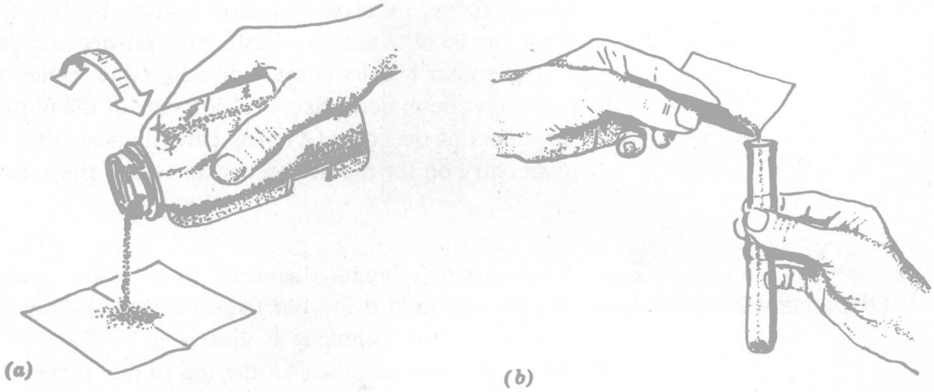


Fig. 1-3 Technique for transferring a solid. (*a*) Slowly rotate the jar until the desired amount of solid falls out. (*b*) Using a creased paper pour the solid into the test tube.

Obtain a square of paper and crease it down the center. Take the paper to the designated shelf or station and obtain a sample of solid by dispensing from a jar. Keep the solid for the next section.

3. Heating Chemicals

Solid and liquid chemicals must be heated with care to prevent explosions and accidents. To heat liquids in beakers or flasks, the container can be placed on a wire gauze supported on an iron ring attached to a ring stand. As shown in Fig. 1-4 the burner can be placed under the gauze and the ring can be adjusted for efficient heating. When heating a liquid in a test tube, hold the tube with your fingers or test tube holder. The test tube holder prevents burned fingers. The test tube should be carefully heated by moving it back and forth in the flame so that the contents are evenly heated. (See Fig. 1-5.) *DANGER: Never* hold the tube in the flame without moving it back and forth and, as soon as it is heated, remove it from the flame. Failure to do this may cause the liquid to suddenly boil and fly out of the tube. Since this can be very dangerous, never point the mouth of the tube at yourself or your neighbor (or the instructor).

Carefully pour the solid on the creased paper into the liquid in the test tube. Light your bunsen burner and heat the contents of the tube in the flame using a test tube holder. Heat the tube for a while but do not let the liquid boil. If it begins to boil, remove from the flame, allow to cool slightly and continue heating. Describe what happens as the contents of the tube are heated (**B**).

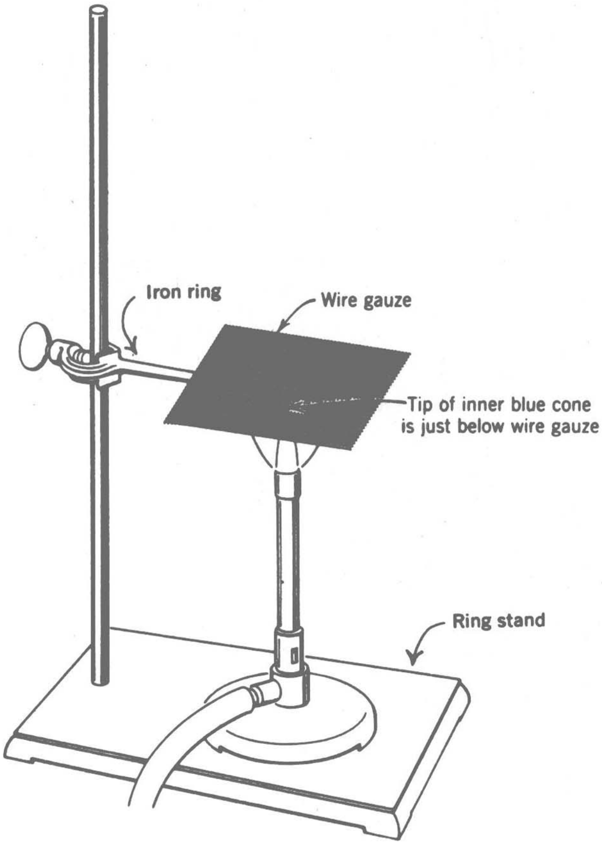


Fig. 1-4 Apparatus for heating liquids. The flask or beaker can be placed on the gauze for heating purposes.

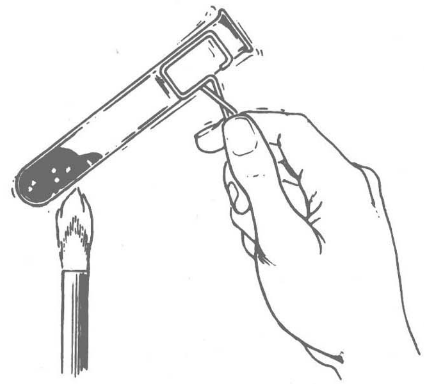


Fig. 1-5 Proper technique for heating a test tube of liquid.

4. Length Measurement

(a) Examine a meter stick and note the parts into which it is subdivided. Answer the following questions regarding the stick. The distance between any two adjacent lines corresponds to what fraction of a meter? ____________(C) To what precision can a length be measured with a meter stick? ____________(D)

(b) Measure the length of a page in your laboratory book first in units of inches and then in units of centimeters.

Length of page ____________ in. ____________ cm (E) Express the inches as a decimal fraction (i.e., $2\frac{1}{2}$ in. = 2.5 in.) ____________ in. (F) Using these two measurements, calculate the number of centimeters in one inch by dividing one by the other. Show your calculations below and express the answer to the correct number of significant digits. (G)

5. Temperature Measurement

Read one of the Celsius thermometers available in the laboratory to the nearest 0.1°C and record the temperature.

Room temperature ____________ (H)

Convert the temperature reading to units of K. ____________ K (I)

Convert the temperature reading to units of °F. ____________ °F (J)

6. Volume Measurement

(a) Examine your 10-ml graduated cylinder. What volume increment can be contained between adjacent lines in the cylinder? (A volume can be read to the nearest 0.1 ml in this cylinder.) (K)

(b) Three partially filled graduated cylinders are on display. For each of these cylinders indicate the volume increment contained between adjacent lines and read the volume of liquid in each as indicated.

Cylinder	Volume increment between lines	Volume		
1000-ml	____________	____________	(to nearest 1 ml)	(L)
100-ml	____________	____________	(to nearest 0.5 ml)	(M)
10-ml	____________	____________	(to nearest 0.1 ml)	(N)

(c) Fill a small test tube with water and pour the water into your 10-ml graduated cylinder to measure the volume of the tube. Record the volume.

Volume of tube ____________ (O)

How full would you have to fill this tube so that you would have about 2 ml of water? (P)

7. Balances and Weighing

The most common type of balance found in the laboratory is the triple beam balance illustrated in Fig. 1-6. This balance is designed to weigh objects to the nearest 0.01 g. The rules for using the balance are:

1. Never place chemicals directly on the balance pan unless you are weighing a piece of inert metal. Always use weighing paper or some container.
2. Never try to make adjustments on the balance. If you have trouble, contact the instructor.
3. Always clean up any chemicals that you spill on the balance or in the weighing area.
4. Always leave the balance with the arrest lever set or so that it is not freely swinging on the knife edge.

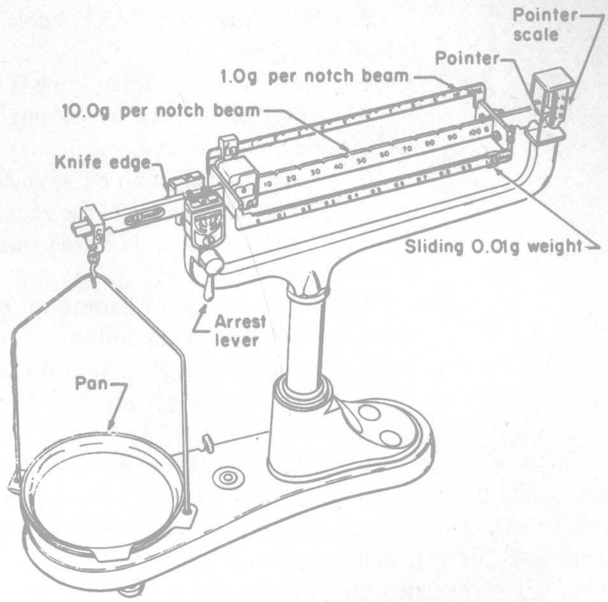


Fig. 1-6 A triple-beam balance.

Use of the Triple Beam Balance. 1. Determine the rest or zero point of the balance by setting the weights of the unloaded balance to zero and allowing the beam to swing freely. As the beam swings, the pointer will move up and down on the pointer scale. As it swings, count the number of division (above the zero) that it moves up and the number of division (below zero) that it moves down. The average of the two counts will give the position at which the pointer would come to rest (the rest point). Make two or three measures of the rest point to be sure. The rest point is used for reference in the weighing operation.

2. Place the object to be weighed on the pan of the balance. The beam will move down to the left. Move the large 10-g weight until the beam moves down to the right. Then move the 10-g weight back one notch. Repeat with the 1-g weight. Once the proper settings of these weights have been found, the sliding 0.01-g weight is moved to the right to a position in which the beam swings freely. At this time, the motion of the pointer on the pointer scale is noted to see if it corresponds to the rest point. If it does not, the sliding mass is moved slightly to the right or left until the motion of the pointer is such that the original rest point is obtained upon averaging of the pointer scale divisions. At this point, the mass is carefully read and recorded. It is always a good idea to double check the reading of the mass.

When a sample of a substance is weighed on a piece of paper or in a container, the mass of the paper or empty container is first determined. Then, the substance is placed on the paper or in the container and the total mass is determined. The mass of the substance is then found by subtracting the mass of the paper or container from the total mass. This is called weighing by difference.

Use of the Top Loader Balance. The top loader balance is another type of balance used in the laboratory. These balances vary in design, but some are built to allow the determination of mass to the nearest 0.01 g.

1. Make sure the balance is level.
2. Turn balance on using the off-on switch.