Chemical Principles in the Laboratory 7th Ed

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Seventh Edition 6

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Preface

A Few Words to the Students

In spite of its many successful theories, chemistry remains, and probably always will remain, an experimental science. As the world of the computer has developed, there are those who feel that one can learn chemistry by working with software and observing reactions on the computer screen rather than in the laboratory. We do not agree with this approach. We believe that there is really no substitute for hands-on laboratory experience if one is to learn what chemistry is all about.

It is not easy to do good experimental work. It requires experience, thought, and care. As beginning students, you have not had much opportunity to do experiments. We feel that the effort you put into your laboratory sessions can pay off in many ways. You can gain a better understanding of how the chemical world works, manual dexterity in manipulating apparatus, an ability to apply mathematics to chemical systems, and, perhaps most importantly, a way of thinking that allows you to better analyze many problems in and out of science. Who knows, perhaps you will find you enjoy doing chemistry and go on to a career as a chemist, as many of our students have.

In writing this manual, we attempted to illustrate many established principles of chemistry with experiments that are as interesting and challenging as possible. These principles are basic to the science, but are usually not intuitively obvious. With each experiment we introduce the theory involved, state in detail the procedures that are used, describe how to draw conclusions from your observations, and, in an Advance Study Assignment, ask you to answer questions similar to those you will encounter in the lab. Before coming to lab, you should read over the experiment for that week, and do the Advance Study Assignment. If you prepare for lab as you should, you will get more out of it. To give an experiment a bit of a challenge, we occasionally ask you to work with chemical unknowns.

Many of you have had considerable experience with computers, using word processors and perhaps scientific software. In the manual there are pages for recording data and making calculations. If you wish, you can prepare your reports with a word processor. You may find that a graphing program is helpful, and may make your calculations on a spreadsheet such as Excel. If you do this, you should record your data in a separate notebook, and do your report and Advance Study Assignment on your word processor.

A Few More Words, This Time to the Teachers

In this, the 7th edition of our manual, we have added a few new experiments, modified a few others, and deleted some that, for one reason or another, did not get used very much in the 6th edition. We relied, as in the past, on the results of questionnaires we sent to some of the schools using that edition, asking for suggestions and comments. Experiment 3 has been modified to improve clarity; Experiment 8 has a new, simpler procedure that does not involve a mercury manometer; Experiment 19 now uses tertiary butyl alcohol as the solvent for freezing point depression; Experiment 39 is new and involves identifying an ionic salt; Experiment 42 is also new and deals with the rate of decomposition of aspirin. We have retained the optional microscale procedures for two experiments, and added an Appendix describing the chemical properties of the cations in Groups I, II and III.

The format of the manual is unchanged, and the order of the experiments makes them compatible with the order of the topics in the text *Chemistry: Principles and Reactions*, Fourth Edition, by William L. Masterton and Cecile N. Hurley. We believe, however, that the overall set of experiments should be appropriate for use with most modern texts in general chemistry.

If this is the first time you are using this manual, we have done what we could to make the transition as easy as possible. The **Instructor's Manual** contains, for each experiment, a list of required equipment and chemicals, the time it will take to do the experiment, and an approximate cost per student. In the second part of the manual we offer comments and suggestions for each experiment that may be helpful, along with some sample data and calculations.

As with any endeavor, there are many people who contribute to the effort. Frank E. DeBoer (North Park College) and Angela Hoffman (University of Portland) offered many helpful comments and suggestions. We also gratefully acknowledge the assistance from the following persons: Melissa Kelly, a Macalester student who helped develop the procedures we are using in the new experiments, and Barbara Ekeberg, who took time from her summer vacation to type the Instructor's Manual. Finally, we would like to thank those who responded to our questionnaire for taking the time to give us their opinions and suggestions:

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It has been a great experience being involved with this manual over its many editions. We have very much appreciated the support of our users, some of whom have been with us over nearly all the editions. We hope that this edition works well for you. We invite any comments, questions and suggestions you may have. Please send them to: Slowinski@Macalester.edu or Wolsey@Macalester.edu.

E. J. Slowinski
W. C. Wolsey
W. L. Masterton

March, 2000

Safety in the Laboratory

Read this section before performing any of the experiments in this manual

A chemistry laboratory can be, and should be, a safe place in which to work. Yet each year in academic and industrial laboratories accidents occur that in some cases injure seriously, or kill, chemists. Most of these accidents could have been foreseen and prevented, had the chemists involved used the proper judgment and taken proper precautions.

The experiments you will be performing have been selected at least in part because they can be done safely. Instructions in the procedures should be followed carefully and in the order given. Sometimes even a change in concentration of one reagent is sufficient to change the conditions of a chemical reaction so as to make it occur in a different way, perhaps at a highly accelerated rate. So, do not deviate from the procedure given in the manual when performing experiments unless specifically told to do so by your instructor.

Eye Protection. One of the simplest, and most important, things you can do in the laboratory to avoid injury is to protect your eyes by routinely wearing safety glasses. Your instructor will tell you what eye protection to use, and you should use it. Goggles worn up on the hair may be attractive, but they are not protective. If you use contact lenses, it is advisable to wear safety glasses as well.

Chemical Reagents. Chemicals in general are toxic materials. This means that they can act as poisons or carcinogens (causes of cancer) if they get into your digestive or respiratory system. Never taste a chemical substance, and avoid getting any chemical on your skin. If that should happen, wash it off promptly with plenty of water. Also, wash your face and hands when you are through working in the laboratory. Do not pipet by mouth; when pipetting, use a rubber bulb or other device to suck up the liquid. Avoid breathing vapors given off by reagents or reactions. If directed to smell a vapor, do so cautiously. Use the hood when the directions call for it.

Some reagents, such as concentrated acids or bases, or bromine, are caustic, which means that they can cause chemical burns on your skin and eat through your clothing. Where such reagents are being used, we note the potential danger with a **CAUTION** sign at that point in the procedure. Be particularly careful when carrying out that step. Always read the label on a reagent bottle before using it; there is a lot of difference between the properties of 1 M H_2SO_4 and those of concentrated (18 M) H_2SO_4.

A few of the chemicals we use are flammable. These include hexane, ethanol, and acetone. Keep your Bunsen burner well away from any open beakers containing such chemicals, and be careful not to spill them on the laboratory bench where they might easily get ignited.

When disposing of the chemical products from an experiment, use good judgment. Some dilute, nontoxic solutions can be poured down the sink and flushed with water. Insoluble or toxic materials should be put in the waste crocks provided for that purpose. Your lab instructor may give you instructions for treatment and disposal of the products from specific experiments.

Safety Equipment. In the laboratory there are various pieces of safety equipment, which may include a safety shower, an eye wash fountain, a fire extinguisher, and a fire blanket. Learn where these items are, so that you will not have to look all over if you ever need them in a hurry.

Laboratory Attire. Come to the laboratory in sensible clothing. Long, flowing robes are out, as are bare feet. Sandals and open-toed shoes offer less protection than regular shoes. Keep long hair tied back, out of the way of flames and reagents.

If an Accident Occurs. During the laboratory course a few accidents will probably occur. For the most part these will not be serious, and might involve a spilled reagent, a beaker of hot water that gets tipped over, a dropped test tube, or a small fire.

A common response in such a situation is panic. A student may respond to an otherwise minor accident by doing something irrational, like running from the laboratory when the remedy for the accident is close at hand. If an accident happens to another student, watch for signs of panic and tell the student what to do; if it seems necessary, help him or her do it. Call the instructor for assistance.

Chemical spills are best handled by washing the area quickly with water from the nearest sink. Use the eye wash fountain if you get something in your eye. In case of a severe chemical spill on your clothing or shoes, use the emergency shower and take off the affected clothing. In case of a fire in a beaker, on a bench, or on your clothing or that of another student, do not panic and run. Smother the fire with an extinguisher, with a blanket, or with water, as seems most appropriate at the time. If the fire is in a piece of equipment or on the lab bench and does not appear to require instant action, have your instructor put the fire out. If you cut yourself on a piece of broken glass, tell your instructor, who will assist you in treating it.

A Message to Foreign Students. Many students from foreign countries take courses in chemistry before they are completely fluent in English. If you are such a student, it may be that in some experiments you will be given directions that you do not completely understand. If that happens, do not try to do that part of the experiment by simply doing what the student next to you seems to be doing. Ask that student, or the instructor, what the confusing word or phrase means, and when you understand what you should do, go ahead. You will soon learn the language well enough, but until you feel comfortable with it, do not hesitate to ask others to help you with unfamiliar phrases and expressions.

Although we have spent considerable time here describing some of the things you should be concerned with in the laboratory from a safety point of view, this does not mean you should work in the laboratory in fear and trepidation. Chemistry is not a dangerous activity when practiced properly. Chemists as a group live longer than other professionals, in spite of their exposure to potentially dangerous chemicals. In this manual we have attempted to describe safe procedures and to employ chemicals that are safe when used properly. Many thousands of students have performed the experiments without having accidents, so you can too. However, we authors cannot be in the laboratory when you carry out the experiments to be sure that you observe the necessary precautions. You and your laboratory supervisor must, therefore, see to it that the experiments are done properly and assume responsibility for any accidents or injuries that may occur.

Contents

Experiment 1

The Densities of Liquids and Solids

One of the fundamental properties of any sample of matter is its density, which is its mass per unit of volume. The density of water is exactly 1.00000 g/cm^3 at 4°C and is slightly less than one at room temperature (0.9970 g/cm^3 at 25°C). Densities of liquids and solids range from values less than that of water to values considerably greater than that of water. Osmium metal has a density of 22.5 g/cm^3 and is probably the densest material known at ordinary pressures.

In any density determination, two quantities must be determined—the mass and the volume of a given quantity of matter. The mass can easily be determined by weighing a sample of the substance on a balance. The quantity we usually think of as "weight" is really the mass of a substance. In the process of "weighing" we find the mass, taken from a standard set of masses, that experiences the same gravitational force as that experienced by the given quantity of matter we are weighing. The mass of a sample of liquid in a container can be found by taking the difference between the mass of the container plus the liquid and the mass of the empty container.

The volume of a liquid can easily be determined by means of a calibrated container. In the laboratory a graduated cylinder is often used for routine measurements of volume. Accurate measurement of liquid volume is made by using a pycnometer, which is simply a container having a precisely definable volume. The volume of a solid can be determined by direct measurement if the solid has a regular geometrical shape. Such is not usually the case, however, with ordinary solid samples. A convenient way to determine the volume of a solid is to measure accurately the volume of liquid displaced when an amount of the solid is immersed in the liquid. The volume of the solid will equal the volume of liquid which it displaces.

In this experiment we will determine the density of a liquid and a solid by the procedure we have outlined. First we weigh an empty flask and its stopper. We then fill the flask completely with water, measuring the mass of the filled stoppered flask. From the difference in these two masses we find the mass of water and then, from the known density of water, we determine the volume of the flask. We empty and dry the flask, fill it with an unknown liquid, and weigh again. From the mass of the liquid and the volume of the flask we find the density of the liquid. To determine the density of an unknown solid metal, we add the metal to the dry empty flask and weigh. This allows us to find the mass of the metal. We then fill the flask with water, leaving the metal in the flask, and weigh again. The increase in mass is that of the added water; from that increase, and the density of water, we calculate the volume of water we added. The volume of the metal must equal the volume of the flask minus the volume of water. From the mass and volume of the metal we calculate its density. The calculations involved are outlined in detail in the Advance Study Assignment.

Experimental Procedure

WEAR YOUR SAFETY GLASSES WHILE PERFORMING THIS EXPERIMENT

A. Mass of a Coin

After you have been shown how to operate the analytical balances in your laboratory, read the section on balances in Appendix IV. Take a coin and measure its mass to 0.0001 g. Record the mass on the Data page. If your balance has a TARE bar, use it to re-zero the balance. Take another coin and weigh it, recording its mass. Remove both coins, zero the balance, and weigh both coins together, recording the total mass. If you have no TARE bar on your balance, add the second coin and measure and record the mass of the two coins. Then remove both coins and find the mass of the second one by itself. When you are satisfied that your results are those you would expect, go to the stockroom and obtain a glass-stoppered flask, which will serve as a pycnometer, and samples of an unknown liquid and an unknown metal.

B. Density of a Liquid

If your flask is not clean and dry, clean it with soap and water, rinse it with a few cubic centimeters of acetone, and dry it by letting it stand for a few minutes in the air or by *gently* blowing compressed air into it for a few moments.

Weigh the dry flask with its stopper on the analytical balance, or the toploading balance if so directed, to the nearest milligram. Fill the flask with distilled water until the liquid level is nearly to the *top* of the ground surface in the neck. Put the stopper in the flask in order to drive out *all* the air and any excess water. Work the stopper gently into the flask, so that it is firmly seated in position. Wipe any water from the outside of the flask with a towel and soak up all excess water from around the top of the stopper.

Again weigh the flask, which should be completely dry on the outside and full of water, to the nearest milligram. Given the density of water at the temperature of the laboratory and the mass of water in the flask, you should be able to determine the volume of the flask very precisely. Empty the flask, dry it, and fill it with your unknown liquid. Stopper and dry the flask as you did when working with the water and then weigh the stoppered flask full of the unknown liquid, making sure its surface is dry. This measurement, used in conjunction with those you made previously, will allow you to find accurately the density of your unknown liquid.

C. Density of a Solid

Pour your sample of liquid from the flask into its container. Rinse the flask with a small amount of acetone and dry it thoroughly. Add small chunks of the metal sample to the flask until the flask is at least half full. Weigh the flask, with its stopper and the metal, to the nearest milligram. You should have at least 50 g of metal in the flask.

Leaving the metal in the flask, fill the flask with water and then replace the stopper. Roll the metal around in the flask to make sure that no air remains between the metal pieces. Refill the flask if necessary, and then weigh the dry, stoppered flask full of water plus the metal sample. Properly done, the measurements you have made in this experiment will allow a calculation of the density of your metal sample that will be accurate to about 0.1%.

DISPOSAL OF REACTION PRODUCTS. Pour the water from the flask. Put the metal in its container. Dry the flask and return it with its stopper and your metal sample, along with the sample of unknown liquid, to the stockroom.

Name ______________________ Section ______________

Experiment 1

Data and Calculations: The Densities of Liquids and Solids

A. Mass of coin 1 __________ g Mass of coin 2 __________ g

Mass of coins 1 and 2 weighed together __________ g
What general law is illustrated by the results of this experiment?

B. Density of unknown liquid

Mass of empty flask plus stopper __________ g

Mass of stoppered flask plus water __________ g

Mass of stoppered flask plus liquid __________ g

Mass of water __________ g

Volume of flask (density of H_2O at 25°C, 0.9970 g/cm^3; at 20°C, 0.9982 g/cm^3) __________ cm^3

Mass of liquid __________ g

Density of liquid __________ g/cm^3

To how many significant figures can the liquid density be properly reported? (See Appendix V.) __________

C. Density of unknown metal

Mass of stoppered flask plus metal __________ g

Mass of stoppered flask plus metal plus water __________ g

Mass of metal __________ g

Mass of water __________ g

Volume of water __________ cm^3

(continued on following page)

Volume of metal ____________ cm^3

Density of metal ____________ g/cm^3

To how many significant figures can the density of the metal be properly reported? ____________

Explain why the value obtained for the density of the metal is likely to have a larger percentage error than that found for the liquid.

Unknown liquid no. ____________ Unknown solid no. ____________

Name ______________________________ **Section** ____________________

Experiment 1

Advance Study Assignment: The Densities of Liquids and Solids

The advance study assignments in this laboratory manual are designed to assist you in making the calculations required in the experiment you will be doing. We do this by furnishing you with sample data and showing in some detail how these data can be used to obtain the desired results. In the advance study assignments we will often include the guiding principles as well as the specific relationships to be employed. If you work through the steps in each calculation by yourself, you should have no difficulty when you are called upon to make the necessary calculations on the basis of the data you obtain in the laboratory.

1. **Finding the volume of a flask.** A student obtained a clean dry glass-stoppered flask. She weighed the flask and stopper on an analytical balance and found the total mass to be 31.692 g. She then filled the flask with water and obtained a mass for the full stoppered flask of 57.826 g. From these data, and the fact that at the temperature of the laboratory the density of water was 0.9973 g/cm³, find the volume of the stoppered flask.

   a. First we need to obtain the mass of the water in the flask. This is found by recognizing that the mass of a sample is equal to the sum of the masses of its parts. For the filled stoppered flask:

   Mass of filled stoppered flask = mass of empty stoppered flask + mass of water, so mass of water = mass of filled flask − mass of empty flask

   Mass of water = ____________ g − ____________ g = ____________ g

   Many mass and volume measurements in chemistry are made by the method used in 1a. This method is called measuring by difference, and is a very useful one.

   b. The density of a pure substance is equal to its mass divided by its volume:

   $$\text{Density} = \frac{\text{mass}}{\text{volume}} \quad \text{or} \quad \text{volume} = \frac{\text{mass}}{\text{density}}$$

   The volume of the flask is equal to the volume of the water it contains. Since we know the mass and density of the water, we can find its volume and that of the flask. Make the necessary calculation.

   Volume of water = volume of flask = ____________ cm³

2. **Finding the density of an unknown liquid.** Having obtained the volume of the flask, the student emptied the flask, dried it, and filled it with an unknown whose density she wished to determine. The mass of the stoppered flask when completely filled with liquid was 54.722 g. Find the density of the liquid.

   a. First we need to find the mass of the liquid by measuring by difference:

   Mass of liquid = ____________ g − ____________ g = ____________ g

(continued on following page)