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CHEMICAL PRINCIPLES

IN THE LABORATORY

ELEVENTH EDITION



Chemical Principles in the Laboratory

Eleventh Edition

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**Chemical Principles in the Laboratory,
Eleventh edition****Emil J. Slowinski, Wayne C. Wolsey,
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Preface

A Few Words to the Students

In spite of its many successful theories, chemistry remains an experimental science. As our world has grown increasingly digital, many initiatives have emerged that pursue the hope that one might learn chemistry by working with software and observing reactions on a computer screen rather than actually carrying them out in a laboratory. While we are proponents of innovation and follow these efforts closely, even dabbling in them ourselves, we remain convinced that chemistry is, at its core, a hands-on laboratory science, in the vast majority of cases best learned by carrying out first-hand experiments with real chemicals. Your instructors must agree, because teaching “wet” laboratory sections is a difficult and expensive undertaking, not the path of least resistance. Please keep in mind that a lot of resources are going into giving you the opportunity to learn chemistry in this way; we urge you to make the most of it!

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. We should make clear, however, that while professional chemists rely on many of the skills that you will learn from the experiments in this manual, the experiments they conduct differ in several important respects. First, the experiments in this manual have all been carried out many times, and their design vetted and refined, such that they offer reasonably reproducible results—at least as much as is possible with an experimental science. In research chemistry, a great deal of thought and effort (and some measure of luck!) must go into the design of experiments if they are to have any hope of producing a reproducible result; we have done that work for you. All of the reagents that go into an experiment must also be correctly prepared and that work will largely be done for you by your instructors or their colleagues. Second, the “correct” outcome of these experiments is known: If something else is obtained (because we can be confident in the experimental design and the reagents used), it is almost always the result of an error made in the course of carrying out the experiment. When doing research-level chemistry, one must always worry about whether the experimental design or some other unknown factor is responsible for an interesting result, and the experiment must be repeated multiple times to ensure that it is reproducible. So even if you find great success in carrying out the experiments in this manual, don’t get too cocky. You will emerge well-prepared to do what professional chemists do . . . but you will not have done it yet!

In writing this manual, we have 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 experiment. 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,” whose identity is unknown to you (but not to your instructors).

This edition includes an appendix entitled “Statistical Treatment of Laboratory Data.” When professional scientists collect experimental data, especially in doing chemical analyses to determine compositions, they usually summarize their results by means of an average (the arithmetic mean) and a measure of how consistent the data are (through a statistical parameter called the standard deviation). In several experiments of this type you will be asked to calculate these quantities from your results. Your instructor may decide to make these calculations an optional part of the experiments, but these values are easily obtained on many standard calculators or via any spreadsheet (such as Excel).

A Few More Words, This Time to the Instructors

While a great deal of this manual remains his handiwork, dating back as far as to its inception in 1969, Professor Emeritus Emil Slowinski has been counseling us on our efforts but is no longer an active participant in revising the manuscript. We miss both his experimental perspicacity and his knack for clear, direct prose aimed at students but have done our humble best to emulate both in putting forth this new edition.

The order of experiments in this laboratory manual makes it compatible with the order of topics in the introductory chemistry text, *Chemistry: Principles and Reactions*, 8th edition, by William Masterton, Cecile N. Hurley, and Edward Neth. We believe, however, that the overall set of experiments should be appropriate for use with most standard texts in general chemistry.

If this is the first time that you are using this manual, we have done what we can to make the transition to a new manual as easy as possible. The *Instructor's Manual*, available online, contains a list of required equipment and chemicals, directions for preparing solutions, and suggestions for dealing with the disposal of chemical waste for each experiment. We also note the time it will take to do the experiments and the approximate cost per student. In the second part of the *Instructor's Manual*, directed to the lab supervisor, we offer comments and suggestions for each experiment that may be helpful, sample data and calculations, and answers to the Advance Study Assignment questions.

As with any endeavor, there are many people who contribute to the effort. We value input and queries from users of the manual, and these have guided many changes in this edition. We would like to thank in particular Arthur Ambrose of Irvine Valley College for his detailed and thoughtful suggestions on units and safety, as well as his colleague Kiana Tabibzadeh, who pointed out a confusing directive in the acetate ion test of Experiment 35, which we hope we have now clarified. Lori Moore (Northeast Iowa Community College) spurred us to find a replacement for mercury in Experiment 37, and Edward Neth and Adam Pangilinan (University of Connecticut–Storrs) suggested the addition of formal data pages to those experiments lacking them. Richard Molinelli (Western Connecticut State University) led us to add guidance on ways to improve the reproducibility of Experiment 14 and to make the data pages for several experiments more logical, while Robert Smith (Tunxis Community College) suggested providing more space for students to show their work on the data pages, a suggestion we have taken to heart in this edition. We appreciate the assistance of many members of the Cengage staff, in the form of editorial and technical support in the preparation of this edition. The cooperation of our chemistry colleagues at Macalester College over the years has also been essential.

It has been a great experience being involved with this laboratory manual over its many editions. We appreciate the support of our users. We encourage any comments, questions, or suggestions you may have. Please send them to wolsey@macalester.edu and rossi@macalester.edu.

Finally, we acknowledge the patient support of our families in the preparation of this edition.

Emil J. Slowinski
Wayne C. Wolsey
Robert C. Rossi

New in This Edition

This edition includes a new OWLv2 LabSkills program that can be bundled with the textbook. LabSkills provides students the opportunity to practice skills and prepare for real laboratory classes in a safe environment. Students work through the assignments in advance of the lab so they can actively rehearse common practical techniques by engaging with simulations, videos and quizzes. Because LabSkills focuses on techniques, instructors can customize assignments to align with the experiments that students will perform in any given lab session. The online course will also offer assessment **created from the Advanced Study Assignments**, and include a MindTap Reader.

The revisions made for this edition focused on improving the manual's usability. All of the experiments have been edited to improve the clarity of the instructions and explanations. Careful attention has been given to the scientific units, terminology, and symbology in order to provide greater consistency and harmony with most general chemistry texts. As in all previous editions, we have changed most of the Advance Study

Assignment questions, particularly those with numerical answers. We have not introduced any new experiments in this edition of *Chemical Principles in the Laboratory*, but we have changed the protocol of Experiment 7, Analysis of an Unknown Chloride. This experiment, which uses the Fajans method with dichlorofluorescein as an adsorption indicator, is classically done using diffuse or reflected light. We have found that identical results can be obtained while using regular ambient laboratory lighting, provided certain precautions are taken, and have revised the instructions accordingly. We have also replaced sulfuric acid with sodium sulfate in Experiment 12, The Alkaline Earths and the Halogens, as it accomplishes the same chemistry more safely than did the strong acid. Since many students now have clocks and even stopwatch functions on their mobile phones, labs involving timing have been revised to include instructions that no longer assume a wall clock is the principal timekeeping device. Formal data pages have been added to Experiments 15 and 19.

As with all chemical educators, we are concerned about the use of mercury and its compounds in the general chemistry laboratory. In Experiment 37, Qualitative Analysis of Group II Cations, we have found an acceptable substitute for the use of mercury(II) chloride in the confirmatory test for tin(II). The organic compound Janus Green is equally able to demonstrate the strong reductive capacity of tin(II). We continue to include Experiment 36, Qualitative Analysis of Group I Cations, for those institutions that favor giving their students hands-on experience with the classic separation and identification steps for silver, lead, and mercury(I). This experiment can also be done in a demonstration setting for institutions who desire to have their students observe the chemical steps. Other than Experiment 36, the 11th edition is “mercury-free.”

Catechol violet was introduced several editions ago as part of the confirmatory test for aluminum in Experiment 38, Qualitative Analysis of Group III Cations, but this was done without providing the basis of the test in the introductory section. The stoichiometry of the aluminum–catechol violet complex has been established (in an IB chemistry project carried out by Kendra Pankow, granddaughter of WCW) to be 1:1, and a complete explanation for the test is now provided in the introduction for Experiment 38.

Several of the experiments in previous editions have assumed all heating would be done with Bunsen burners. We have now rewritten the directions for several of these experiments such that they are agnostic to the use of either hot plates or Bunsen burners in the heating steps. Experiment 9, Molar Mass of a Volatile Liquid, poses a potential fire hazard when conducted with Bunsen burners, and is best done using hot plates; it has been rewritten to assume this to be the case. A few other experiments which necessitate the use of high temperatures, such as those involving the heating of crucibles, or support of combustion tests, remain designed around the use of Bunsen burners.

The sections on electrochemistry have been revised to allude to the complexities involved in quantitative electrochemical measurements (without making the discussion too hard to follow) and to make the terminology used more precise.

Appendix VII has been updated to work with a broader range of Excel versions, while Appendix III was abridged (the unstable elements removed) and its molar mass values updated. Readability improvements were made to the table format of Appendix II.

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 even kill, chemists. Most of these accidents could have been foreseen and prevented had the chemists involved used 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 the 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. Glasses worn up on the hair may look hip, but they will not protect your eyes. If you use contact lenses, it is even more critical that you 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 contact should occur, wash off the affected area promptly with plenty of water. Also, wash your face and hands when you are through working in the laboratory. Never 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 a fume hood when the directions call for it.

Some reagents, such as concentrated acids or bases, or bromine, are corrosive, 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:** box 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 heptane, ethanol, and acetone. Keep ignition sources, like hotplates and Bunsen burners, well away from any open beakers containing such chemicals, and be careful not to spill them on the laboratory bench where they might be easily ignited.

When disposing of the chemical products from an experiment, use good judgment. Some dilute, nontoxic solutions can be poured down the sink, flushing with plenty of water. Insoluble or toxic materials should be put in the waste containers 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 emergency eye wash, a fire extinguisher, and a fire blanket. Learn where these items are, so that you will not have to waste precious moments looking for them 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. Tight-fitting jewelry (especially rings) can exacerbate the harm caused by chemical exposure and should not be worn in lab.

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 a mishap befalls a fellow 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.

Most chemical spills are best handled by quickly absorbing wet material with a paper towel and then washing the area 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 these 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.

Disclaimer: Chemistry experiments employing the use of chemicals and laboratory equipment can be dangerous, and misuse may cause serious bodily injury. Cengage Learning encourages you to speak with your instructor and/or laboratory manager and become acquainted with your school's laboratory safety regulations before attempting any experiments. Cengage Learning and the Authors have provided, for your convenience only, safety information intended to serve as a starting point for good practices. Cengage Learning and the Authors make no guarantee or representations as to the accuracy or sufficiency of such information and/or instructions.

Contents

Experiment 1

The Densities of Liquids and Solids 1

Experiment 2

Resolution of Matter into Pure Substances, I. Paper Chromatography 7

Experiment 3

Resolution of Matter into Pure Substances, II. Fractional Crystallization 15

Experiment 4

Determination of a Chemical Formula 23

Experiment 5

Identification of a Compound by Mass Relationships 29

Experiment 6

Properties of Hydrates 35

Experiment 7

Analysis of an Unknown Chloride 41

Experiment 8

Verifying the Absolute Zero of Temperature—Determination of the Atmospheric Pressure 47

Experiment 9

Molar Mass of a Volatile Liquid 57

Experiment 10

Analysis of an Aluminum-Zinc Alloy 63

Experiment 11

The Atomic Spectrum of Hydrogen 71

Experiment 12

The Alkaline Earths and the Halogens—Two Families in the Periodic Table 81

Experiment 13

The Geometrical Structure of Molecules—An Experiment Using Molecular Models 89

Experiment 14

Heat Effects and Calorimetry **99**

Experiment 15

The Vapor Pressure and Heat of Vaporization of a Liquid **107**

Experiment 16

The Structure of Crystals—An Experiment Using Models **117**

Experiment 17

Classification of Chemical Substances **129**

Experiment 18

Some Nonmetals and Their Compounds—Preparations and Properties **137**

Experiment 19

Molar Mass Determination by Depression of the Freezing Point **145**

Experiment 20

Rates of Chemical Reactions, I. The Iodination of Acetone **153**

Experiment 21

Rates of Chemical Reactions, II. A Clock Reaction **163**

Experiment 22

Properties of Systems in Chemical Equilibrium—Le Châtelier's Principle **173**

Experiment 23

Determination of the Equilibrium Constant for a Chemical Reaction **185**

Experiment 24

The Standardization of a Basic Solution and the Determination of the Molar Mass of an Acid **195**

Experiment 25

pH Measurements—Buffers and Their Properties **203**

Experiment 26

Determination of the Solubility Product of $\text{Ba}(\text{IO}_3)_2$ **213**

Experiment 27

Relative Stabilities of Complex Ions and Precipitates Prepared from Solutions of Copper(II) **221**

Experiment 28

Determination of the Hardness of Water **229**

- Experiment 29**
Synthesis and Analysis of a Coordination Compound **235**
- Experiment 30**
Determination of Iron by Reaction with Permanganate—A Redox Titration **245**
- Experiment 31**
Determination of an Equivalent Mass by Electrolysis **251**
- Experiment 32**
Voltaic Cell Measurements **259**
- Experiment 33**
Preparation of Copper(I) Chloride **269**
- Experiment 34**
Development of a Scheme for Qualitative Analysis **275**
- Experiment 35**
Spot Tests for Some Common Anions **281**
- Experiment 36**
Qualitative Analysis of Group I Cations **289**
- Experiment 37**
Qualitative Analysis of Group II Cations **297**
- Experiment 38**
Qualitative Analysis of Group III Cations **305**
- Experiment 39**
Identification of a Pure Ionic Solid **313**
- Experiment 40**
The Ten Test Tube Mystery **321**
- Experiment 41**
Preparation of Aspirin **329**
- Experiment 42**
Rate Studies on the Decomposition of Aspirin **337**
- Experiment 43**
Analysis for Vitamin C **345**

Appendix I

Vapor Pressure and Density of Liquid Water **351**

Appendix II

Summary of Solubility Properties of Ions and Solids **352**

Appendix IIA

Some Properties of the Cations in Groups I, II, and III **355**

Appendix III

Standard Atomic Weights of the Elements (Scaled relative to Carbon-12 = 12 g/mol) **359**

Appendix IV

Making Measurements—Laboratory Techniques **361**

Appendix V

Mathematical Considerations—Significant Figures and Making Graphs **371**

Appendix VI

Suggested Locker Equipment **377**

Appendix VII

Introduction to Excel **379**

Appendix VIII

Statistical Treatment of Laboratory Data **383**

Experiment 1

The Densities of Liquids and Solids

Given a sample of a pure liquid, we can measure many of its characteristics. Its temperature, mass, color, and volume are among the many properties we can determine. We find that if we measure the mass and volume of different samples of the liquid, the mass and volume of each sample are related in a simple way: if we divide the mass by the volume, the result we obtain is the same for each sample, independent of its mass. That is, for samples A, B, and C of the liquid at constant temperature and pressure,

$$\text{Mass}_A/\text{Volume}_A = \text{Mass}_B/\text{Volume}_B = \text{Mass}_C/\text{Volume}_C = \text{a constant}$$

That constant, which is clearly independent of the size of the sample, is called its **density** and is one of the fundamental properties of the liquid. The density of water is exactly 1.00000 g/cm^3 at 4°C and is slightly less than that at room temperature (0.9970 g/cm^3 at 25°C). Densities of liquids and solids range from values that are less than that of water to values that are much greater. 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 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 geometric shape. Such is not usually the case, however, with ordinary solid samples. A convenient way to determine the volume of a solid is to accurately measure 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 the liquid that 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.

WEAR YOUR SAFETY GLASSES WHILE
PERFORMING THIS EXPERIMENT

Experimental Procedure

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, 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 detergent solution and water, rinse it with a few milliliters 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 an analytical balance, or a top-loading 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 (see Appendix I). 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 accurately determine 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.

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 part of the 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

Temperature in the laboratory _____ °C

Volume of flask (obtain density of water from Appendix I)
_____ mL

Mass of liquid _____ g

Density of liquid _____ g/mL

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

(continued on following page)

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 _____ mL

Volume of metal _____ cm³

Density of metal _____ g/cm³

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 # _____ Unknown solid # _____

Experiment 1**Advance Study Assignment: Densities of Solids and Liquids**

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 that 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 34.166 g. She then filled the flask with water and obtained a mass for the full stoppered flask of 68.090 g. From these data, and the fact that at the temperature of the laboratory the density of water was 0.9975 g/mL, 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

$$\text{Mass of water} = \text{_____ g} - \text{_____ g} = \text{_____ g}$$

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.

$$\text{Volume of water} = \text{volume of flask} = \text{_____ mL}$$

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 liquid whose density she wished to determine. The mass of the stoppered flask when completely filled with liquid was 57.418 g. Find the density of the liquid.

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

$$\text{Mass of liquid} = \text{_____ g} - \text{_____ g} = \text{_____ g}$$

(continued on following page)

6 Experiment 1 The Densities of Liquids and Solids

- b. Since the volume of the liquid equals that of the flask, we know both the mass and volume of the liquid and can easily find its density using the equation in 1b. Make the calculation.

$$\text{Density of liquid} = \text{_____ g/mL}$$

3. Finding the density of a solid.

The student then emptied the flask and dried it once again. To the empty flask she added pieces of a metal until the flask was about three-fourths full. She weighed the stoppered flask and its metal contents and found that the mass was 116.150 g. She then filled the flask with water, stoppered it, and obtained a total mass of 119.827 g for the flask, stopper, metal, and water. Find the density of the metal.

- a. To find the density of the metal we need to know its mass and volume. We can easily obtain its mass by the method of differences:

$$\text{Mass of metal} = \text{_____ g} - \text{_____ g} = \text{_____ g}$$

- b. To determine the volume of metal, we note that the volume of the flask must equal the volume of the metal plus the volume of water in the filled flask containing both metal and water. If we can find the volume of water, we can obtain the volume of metal by the method of differences. To obtain the volume of the water we first calculate its mass:

$$\text{Mass of water} = \text{mass of (flask + stopper + metal + water)} - \text{mass of (flask + stopper + metal)}$$

$$\text{Mass of water} = \text{_____ g} - \text{_____ g} = \text{_____ g}$$

The volume of water is found from its density, as in 1b. Note that 1 mL = 1 cm³. Make the calculation.

$$\text{Volume of water} = \text{_____ mL} = \text{_____ cm}^3$$

- c. From the volume of the water, we calculate the volume of metal:

$$\text{Volume of metal} = \text{volume of flask} - \text{volume of water}$$

$$\text{Volume of metal} = \text{_____ cm}^3 - \text{_____ cm}^3 = \text{_____ cm}^3$$

From the mass and volume of metal, we find the density, using the equation in 1b. Make the calculation.

$$\text{Density of metal} = \text{_____ g/cm}^3$$

Now go back to Question 1 and check to see that you have reported the proper number of significant figures in each of the results you calculated in this assignment. Use the rules on significant figures as given in your chemistry text or Appendix V.

Experiment 2

Resolution of Matter into Pure Substances, I. Paper Chromatography

The fact that different substances have different solubilities in a given solvent can be used in several ways to effect a separation of substances from mixtures in which they are present. We will see in an upcoming experiment how fractional crystallization allows us to obtain pure substances by relatively simple procedures based on solubility properties. Another widely used resolution technique, which also depends on solubility differences, is chromatography.

In a chromatographic experiment a mixture is deposited on some solid adsorbing substance, which might consist of a strip of filter paper, a thin layer of silica gel on a piece of glass, some finely divided charcoal packed loosely in a glass tube, or even a resin coating the walls of an incredibly small-diameter tube.

The components of a mixture are adsorbed on a solid to varying degrees, depending on the nature of the component, the nature of the adsorbent, and the temperature. A solvent is then made to flow through the adsorbent solid under applied or gravitational pressure, or by the capillary effect. As the solvent passes the deposited sample, the various components tend, to varying extents, to be dissolved and swept along the solid. The rate at which a component will move along the solid depends on its relative tendency to be dissolved in the solvent as opposed to adsorbed on the solid. The net effect is that, as the solvent passes slowly through the solid, the components separate from each other and move along as rather diffuse zones. With the proper choice of solvent and adsorbent, it is possible to resolve many complex mixtures by this procedure. If necessary, we can usually recover a given component by identifying the position of the zone containing the component, removing that part of the solid from the system, and eluting the desired component with a suitable (good) solvent.

The name given to a particular kind of chromatography depends upon the manner in which the experiment is conducted. Thus, we have column, thin-layer, paper, and gas chromatography, all in very common use (Fig. 2.1). Chromatography in its many possible variations offers the chemist one of the best methods, if not the best method, for resolving a mixture into pure substances, regardless of whether that mixture

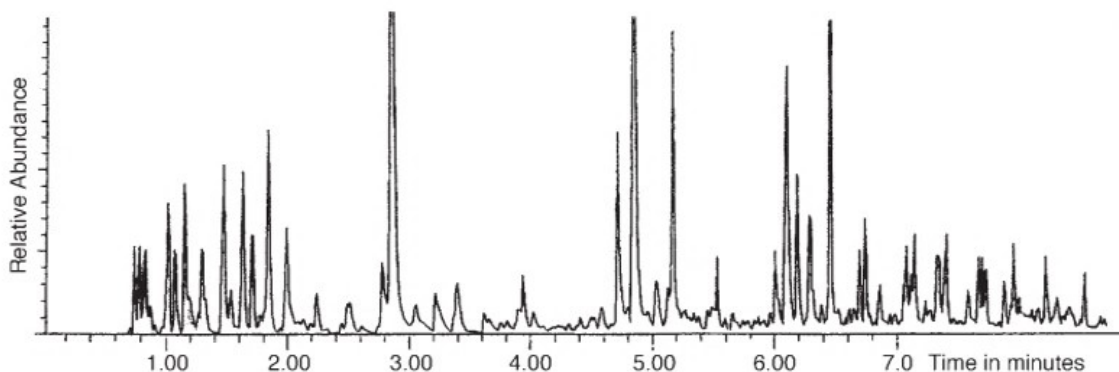


Figure 2.1 This is a gas chromatogram of a sample of unleaded gasoline. Each peak corresponds to a different molecule, so gasoline has many components: at least 50, each of which can be identified. The molar masses vary from about 50 to about 150, with the largest peak, at about 3 minutes, being due to toluene, $C_6H_5CH_3$. The sample size for this chromatogram was less than 10^{-6} grams, <0.001 mg! Gas chromatography offers the best method for resolution of complex volatile mixtures. Chromatogram courtesy of Prof. Becky Hoyer at Macalester College.

consists of a gas, a volatile liquid, or a group of nonvolatile, relatively unstable, complex organic compounds.

In this experiment we will use paper chromatography to separate a mixture of metallic ions in solution. A sample containing a few micrograms of ions is applied as a spot near one edge of a piece of filter paper. That edge is immersed in a solvent, with the paper held vertically. As the solvent rises up the paper by capillary action, it will carry the metallic ions along with it to a degree that depends upon the relative tendency of each ion to dissolve in the solvent rather than adsorb on the paper. Because the ions differ in their properties, they move at different rates and become separated on the paper. The position of each ion during the experiment can be recognized if the ion is colored, as some of them are. At the end of the experiment their positions are established more clearly by treating the paper with a staining reagent that reacts with each ion to produce a colored product. By observing the position and color of the spot produced by each ion, and the positions of the spots produced by an unknown mixture containing some of those ions, you can readily determine the ions present in the unknown.

It is possible to describe the position of spots such as those you will be observing in terms of a quantity called the R_f ("Retention factor") value. In the experiment the solvent rises a certain distance, say L centimeters. At the same time a given component will usually rise a smaller distance, say D centimeters. The ratio of D/L is called the R_f value for that component:

$$R_f = \frac{D}{L} = \frac{\text{distance component moves}}{\text{distance solvent moves}} \quad (1)$$

The R_f value is a characteristic property of a given component in a chromatographic experiment conducted under particular conditions. Within some limits, it does not depend upon concentration or upon the other components present. Hence, it can be reported in the literature and used by other researchers doing similar analyses. In the experiment you will be doing, you will be asked to calculate the R_f values for each of the cations studied.

Experimental Procedure

WEAR YOUR SAFETY GLASSES WHILE PERFORMING THIS EXPERIMENT



Obtain an unknown mixture and a piece of filter paper about 19 cm long and 11 cm wide. Along the 19-cm edge, draw a pencil line about 1 cm from that edge. Starting 1.5 cm from the end of the line, mark the line at 2-cm intervals. Label the segments of the line as shown in Figure 2.2, with the formulas of the ions to be studied and the known and unknown mixtures.

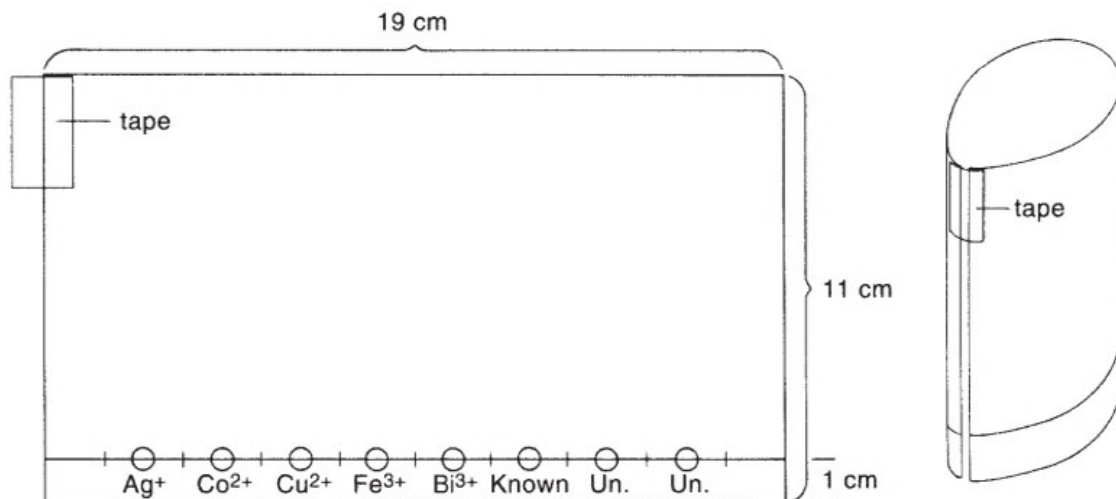


Figure 2.2

Put two or 3 drops of 0.1 M solutions of the following compounds in separate micro test tubes, one solution to a tube:



In solution these substances exist as ions. The metallic cations are Ag^+ , Co^{2+} , Cu^{2+} , Fe^{3+} , and Bi^{3+} , respectively. One drop of each solution contains about 50 micrograms of cation. Into a sixth micro test tube put 2 drops of each of the five solutions; swirl until the solutions are well mixed. This mixture will be our known, since we know it contains all of the cations.

Your instructor will furnish you with a fine capillary tube, which will serve as an applicator. Test the application procedure by dipping the applicator into one of the colored solutions and touching it momentarily to a round piece of filter paper. The liquid from the applicator should form a spot no larger than 8 mm in diameter. Practice making spots until you can reproduce the spot size each time.

Clean the applicator by dipping it about 1 cm into distilled water and then touching the round filter paper to remove the liquid. In this case continue to hold the applicator against the paper until all the liquid in the tube is gone. Repeat the cleaning procedure one more time. Dip the applicator into one of the cation solutions and put a spot on the line on the rectangular filter paper in the region labeled for that cation. Clean the applicator twice, and repeat the procedure with another solution. Continue this approach until you have put a spot for each of the five cations and the known and unknown mixtures on the paper, cleaning the applicator between solutions. Dry the paper by moving it in the air or holding it briefly in front of a hair dryer or heat lamp (low setting). Apply the known and unknown mixtures three more times to the same spots; the known and unknown mixtures are less concentrated than the cation solutions, so this procedure will increase the amount of each ion present in the spots. Be sure to dry the spots between applications, since otherwise they will get larger. Don't heat the paper more than necessary, just enough to dry the spots.

Draw about 15 mL of eluting solution from the supply on the reagent shelf. This solution is made by mixing a solution of hydrochloric acid (HCl) in water, with ethanol and butanol, which are organic solvents. Pour the eluting solution into a 600-mL beaker and cover with a watch glass.

Check to make sure that the spots on the filter paper are all dry. Place a 4- to 5-cm length of tape along the upper end of the left edge of the paper, as shown in Figure 2.2, so that about half of the tape is on the paper. Form the paper into a cylinder by attaching the tape to the other edge in such a way that the edges are parallel but do not overlap. When you are finished, the pencil line at the bottom of the cylinder should form a circle (approximately, anyway) and the two edges of the paper should not quite touch. Stand the cylinder up on the lab bench to check that such is the case and readjust the tape if necessary. *Do not* tape the lower edges of the paper together.

Place the cylinder in the eluting solution in the 600-mL beaker, with the sample spots down near the liquid surface. The paper should not touch the walls of the beaker. Cover the beaker with the watch glass. The solvent will gradually rise by capillary action up the filter paper, carrying along the cations at different rates. After the process has gone on for a few minutes, you should be able to see colored spots on the paper, showing the positions of some of the cations.

While the experiment is proceeding, you can test the effect of the staining reagent on the different cations. Put an 8-mm spot of each of the cation solutions on a clean piece of round filter paper, labeling each spot and cleaning the applicator between solutions. Dry the spots as before. Some of them will have a little color; record those colors on the data page. Put the filter paper on a paper towel, and, using the spray bottle on the lab bench, spray the paper evenly with the staining reagent, getting the paper moist but not really wet. The staining reagent is a solution containing potassium ferrocyanide and potassium iodide. This reagent forms colored precipitates or reaction products with many cations, including all of those used in this experiment. Note the colors obtained with each of the cations. Considering that each spot contains less than 50 micrograms of cation, the tests are quite definitive.

When the eluting solution has risen to within about 2 cm of the top of the filter paper (it will take about 75 minutes), remove the cylinder from the beaker and take off the tape. Draw a pencil line along the solvent front. Dry the paper with gentle heat until it is quite dry. Note any cations that must be in your unknown by virtue of your being able to see their colors. Then, with the filter paper set out flat on a paper towel, spray it as before

10 Experiment 2 Resolution of Matter into Pure Substances, I. Paper Chromatography

with the staining reagent. Any cations you identified in your unknown before staining should be observed, as well as any that require staining for detection.

Measure the distance from the straight line on which you applied the spots to the solvent front, which is distance L in Equation 1. Then measure the distance from the pencil line to the center of the spot made by each of the cations, when pure and in the known; this is distance D . Calculate the R_f value for each cation. Then calculate R_f values for the cations in the unknown. How do the R_f values compare?

DISPOSAL OF REACTION PRODUCTS. When you are finished with the experiment, pour the eluting solution into the waste container, not down the sink. Wash your hands before leaving the laboratory.

Name _____ Section _____

Experiment 2

Data and Calculations: Resolution of Matter into Pure Substances, I. Paper Chromatography

	Ag ⁺	Co ²⁺	Cu ²⁺	Fe ³⁺	Bi ³⁺
Colors (if observed)					
Dry	_____	_____	_____	_____	_____
After staining	_____	_____	_____	_____	_____
Pure Cations					
Distance solvent moved (<i>L</i>)	_____	_____	_____	_____	_____
Distance cation moved (<i>D</i>)	_____	_____	_____	_____	_____
 <i>R_f</i>	_____	_____	_____	_____	_____
Known Mixture					
Distance solvent moved	_____	_____	_____	_____	_____
Distance cation moved	_____	_____	_____	_____	_____
 <i>R_f</i>	_____	_____	_____	_____	_____

(continued on following page)

Unknown Mixture

Cations identified

Dry _____

After staining _____

Distance solvent moved _____

Distance cation moved _____

R_f _____

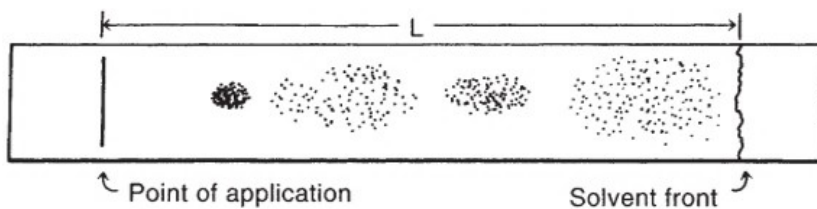
Composition of unknown _____

Unknown # _____

Experiment 2

Advance Study Assignment: Resolution of Matter into Pure Substances, I. Paper Chromatography

1. A student chromatographs a mixture, and after developing the spots with a suitable reagent he observes the following:



What are the R_f values?

2. Explain, in your own words, why samples can often be separated into their components by chromatography.

14 **Experiment 2** Resolution of Matter into Pure Substances, I. Paper Chromatography

3. The solvent moves 3 cm in about 10 minutes. Why shouldn't the experiment be stopped at that time instead of waiting 75 minutes for the solvent to move 10 cm?

4. In this experiment it takes about 10 microliters of solution to produce a spot 1 cm in diameter. If the $\text{Bi}(\text{NO}_3)_3$ solution contains about 21 g Bi^{3+} per liter, how many micrograms of Bi^{3+} ion are there in one spot?

_____ micrograms

Experiment 3

Resolution of Matter into Pure Substances, II. Fractional Crystallization

One of the important problems faced by chemists is that of determining the nature and state of purity of the substances with which they work. In order to perform meaningful experiments, chemists must ordinarily use essentially pure substances, which are often prepared by separation from complex mixtures.

In principle the separation of a mixture into its component substances can be accomplished by carrying the mixture through one or more physical changes, experimental operations in which the nature of the components remains unchanged. Because the physical properties of various pure substances are different, physical changes frequently allow an enrichment of one or more substances in one of the fractions that is obtained during the change. Many physical changes can be used to accomplish the resolution of a mixture, but in this experiment we will restrict our attention to one of the simpler ones in common use, namely, fractional crystallization.

The solubilities of solid substances in different kinds of liquid solvents vary widely. Some substances are essentially insoluble in all known solvents; the materials we classify as macromolecular are typical examples. Most materials are noticeably soluble in one or more solvents. Those substances that we call salts often have very appreciable solubility in water but relatively little solubility in other liquids. Organic compounds, whose molecules contain carbon and hydrogen atoms as their main constituents, are often soluble in organic liquids such as benzene or carbon tetrachloride.

We also often find that the solubility of a given substance in a liquid is sharply dependent on temperature. Most substances are more soluble in a given solvent at high temperatures than at low temperatures, although there are some materials whose solubility is practically temperature-independent and a few others that become less soluble as temperature increases.

By taking advantage of the differences in solubility of different substances, we often find it possible to isolate the components of a mixture in essentially pure form.

In this experiment you will be given a sample containing silicon carbide, potassium nitrate, and copper sulfate. Your goal will be to separate two pure components from the mixture, using water as the solvent. Silicon carbide, SiC, is a black, very hard material; it is the classic abrasive, and completely insoluble in water. Potassium nitrate, KNO_3 , and copper sulfate, $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$, are water-soluble ionic substances, with different solubilities at different temperatures, as indicated in Figure 3.1. The copper sulfate we will use is blue as a crystalline hydrate as well as in solution. Its solubility increases fairly rapidly with temperature. Potassium nitrate is a white solid, clear and colorless in solution. Its solubility in water increases about 20-fold between 0°C and 100°C .

Given a mixture containing roughly equal amounts of SiC and KNO_3 and a small amount of $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$, we separate out the silicon carbide first. This is done by simply stirring the mixture with water, which dissolves all of the potassium nitrate and copper sulfate in the mixture. The insoluble silicon carbide remains behind and is filtered off.

The solution obtained after filtration contains KNO_3 and CuSO_4 in a rather large amount of water. Some of the water is removed by boiling, and then the solution is cooled to 0°C . At that point the KNO_3 is not very soluble, and most of it crystallizes from solution. Since CuSO_4 is not present in large amount, its solubility is not exceeded and it remains in solution. The solid KNO_3 is separated from the solution by filtration. This procedure, by which a substance can be separated from an impurity, is called fractional crystallization.

The solid potassium nitrate one recovers is contaminated by a small amount of copper sulfate. The purity of the solid can be markedly increased by stirring it with a small amount of water and then filtering off the dissolved CuSO_4 . The purity can be established by the intensity of the color produced by the copper impurity when treated with ammonia, NH_3 .

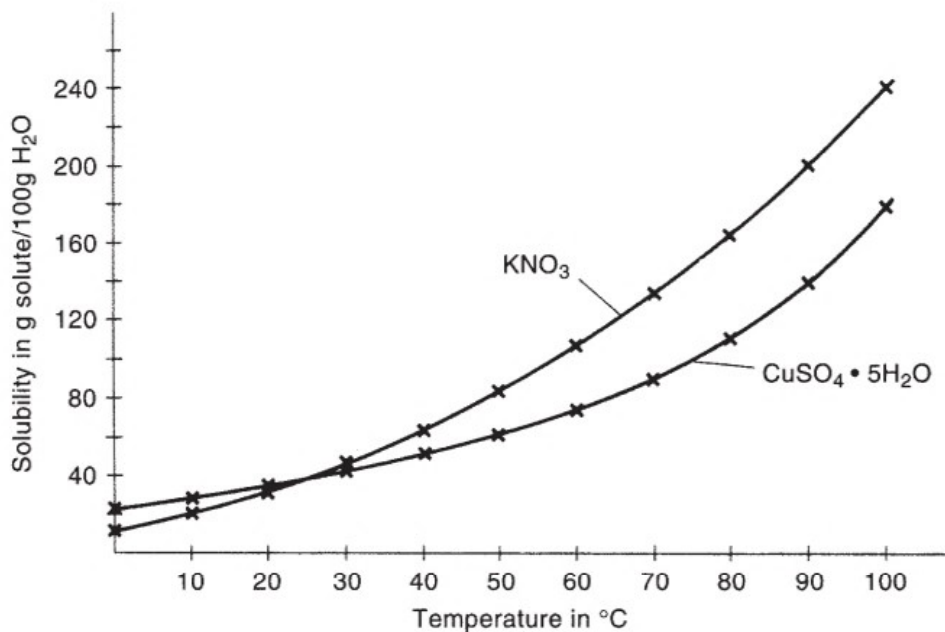


Figure 3.1

Experimental Procedure

WEAR YOUR SAFETY GLASSES WHILE
PERFORMING THIS EXPERIMENT



Obtain a Buchner funnel, a suction flask, and a sample (about 20 grams) of your unknown solid mixture.

Weigh a 150-mL beaker, a 50-mL beaker, and a piece of filter paper, one item at a time, on a top-loading balance. These, and all other weighings in this experiment, should be made to ± 0.1 g. (Do not use an analytical balance for any weighings.) Use the masses you obtain as needed in your calculations.

Add your sample to the 150-mL beaker and weigh again. Then add about 40 mL of distilled water, which will be enough to dissolve the soluble solids. Light your Bunsen burner and adjust the flame so it is blue, quiet, and of moderate size.

Separation of SiC

Support the beaker with its contents on a piece of wire gauze on an iron ring. Warm it gently to about 50°C , while stirring the mixture. When the blue and white solids are all dissolved, pour the contents of the beaker into a Buchner funnel while gentle suction is being applied (see Fig. 3.2 and Appendix IV). Transfer as much of the black solid carbide as you can to the funnel, using your rubber policeman. Transfer the blue filtrate to the (cleaned) 150-mL beaker and add 15 drops of 6 M nitric acid, HNO_3 , which will help ensure that the copper sulfate remains in solution in later steps. Reassemble the funnel, apply suction, and wash the SiC on the filter paper with distilled water. Continue the suction for a few minutes to dry the SiC. Turn off the suction, and, using your spatula, lift the filter paper and the SiC crystals from the funnel, and put the paper on the lab bench so that the crystals may dry in the air. When you are finished with the rest of the experiment, weigh the dry SiC on its piece of filter paper.

Separation of KNO_3

Heat the blue filtrate in the beaker to the boiling point, and then boil gently until the white crystals of KNO_3 are visible in the liquid.

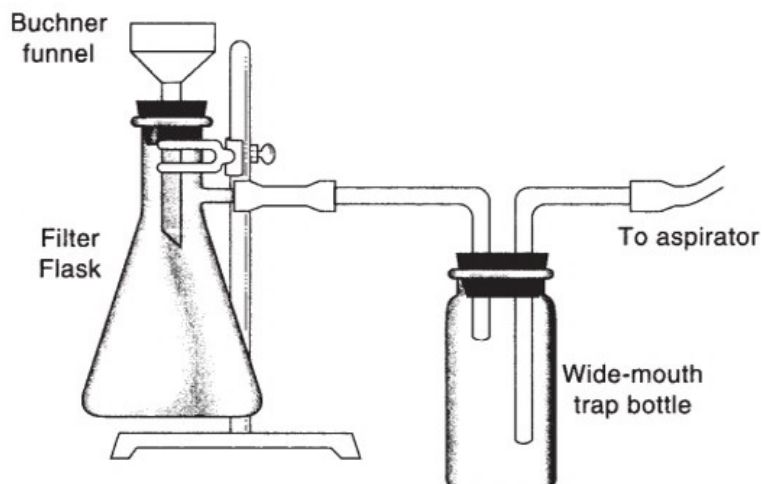


Figure 3.2 To operate the Buchner funnel, put a piece of circular filter paper in the funnel. Thoroughly moisten the filter paper with distilled water from a wash bottle, then immediately turn on the suction. Keep the suction on while filtering the sample.

CAUTION: *The hot liquid may have a tendency to bump, so do not heat it too strongly.*

While the filtrate is being heated, prepare some ice-cold distilled water by putting your wash bottle in an ice-water bath.

When white crystals are clearly apparent in the boiling liquid (the solution may appear cloudy at that point), stop heating and add 12 mL distilled water to the solution. Stir the mixture with a glass stirring rod to dissolve the solids, including any on the walls; if necessary, warm the solution, but do not boil it.

Cool the solution to room temperature in a water bath, and then to about 0°C in an ice bath. White crystals of KNO_3 will come out of solution. Stir the cold slurry of crystals for several minutes. Check the temperature of the slurry with your thermometer. It should be no more than 3°C. Continue stirring until your mixture gets to that temperature or even a bit lower.

Assemble the Buchner funnel. Chill it by adding about 100 mL of ice-cold distilled water from your wash bottle, and, after about a minute, by drawing the water through with suction. Filter the KNO_3 slurry through the cold Buchner funnel. Your rubber policeman will be helpful when you transfer the last of the crystals. Press the crystals dry with a clean piece of filter paper, and continue to apply suction for another 30 seconds. Turn off the suction. Lift the filter paper and the crystals from the funnel, and put the paper and crystals on the lab bench.

DISPOSAL OF REACTION PRODUCTS. By this procedure you have separated most of the KNO_3 in your sample from the CuSO_4 , which is present in the solution in the suction flask. This solution may now be discarded, so dispose of it as directed by your instructor.

Clean and dry your 150-mL beaker and to it add the KNO_3 crystals from the filter paper. Weigh the beaker and its contents.

Analysis of the Purity of the KNO_3

The KNO_3 crystals you have prepared contain a small amount of CuSO_4 as an impurity. To find the amount of CuSO_4 present, weigh out 0.5 g of the crystals into your weighed 50-mL beaker. Dissolve the crystals in 3 mL distilled water, and then add 3 mL 6 M ammonia (NH_3). The copper impurity will form a blue solution in the NH_3 . Pour the solution into a small (13 × 100 mm) test tube. Compare the intensity of the blue color with that in a series of standard solutions prepared by your instructor. Estimate the relative concentration of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ in your product.