CENGAGE LABORATORY SERIES *for*
General Chemistry

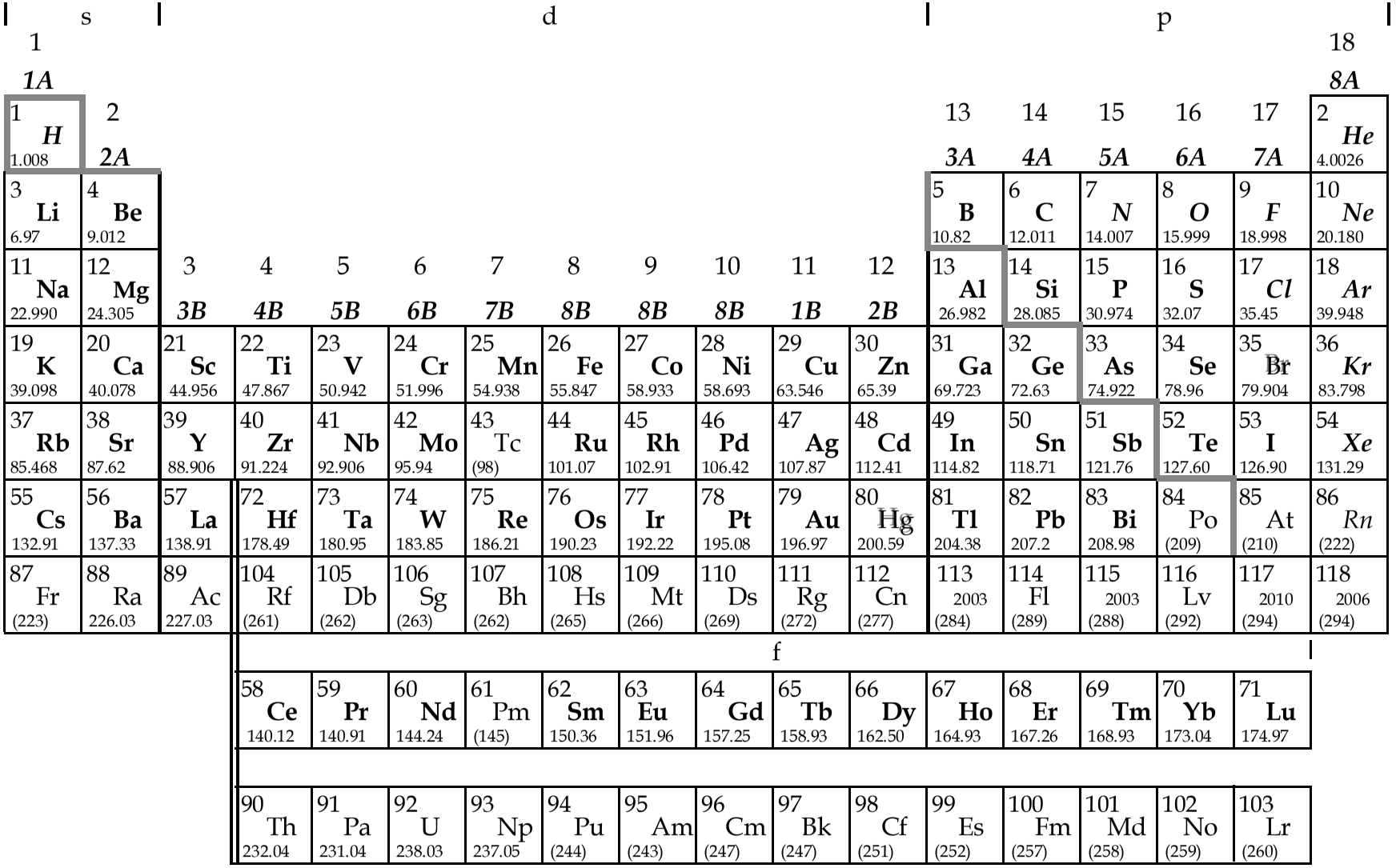
Experiments in

General Chemistry

SIXTH EDITION

Steven L. Murov

PERIODIC TABLE OF THE ELEMENTS



key: bold or normal italics - gas, shadow - liquid, bold or normal - solid, normal print - all known isotopes are radioactive

Experiments in General Chemistry

SIXTH EDITION

Steven L. Murov
Modesto Junior College

CENGAGE Learning

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***Experiments in General Chemistry*, 6e**
Steven L. Murov

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Text and Cover Designer: PreMediaGlobal
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Cengage Learning
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EXPERIMENTS IN GENERAL CHEMISTRY

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^{C} indicates that a centigram balance will suffice for this experiment.
^{M} indicates that a milligram balance is recommended for this experiment.

EXPERIMENTS IN GENERAL CHEMISTRY

PREFACE

A collection of the best short works of the late, great physicist, Richard Feynman, is entitled *The Pleasure of Finding Things Out.* The goal of this text is to give students the opportunity to experience the pleasure of finding things out. Curiosity is a wonderful attribute that motivates humans to use creative talents to probe, explore and explain the mysteries of our universe. The search for explanations for the mysteries provides an adventure that helps to make life exciting and rewarding. The pathways to the solutions often lead to valuable insights and unexpected significant discoveries. This text has been designed to provide a stimulating environment that will promote curiosity and motivate students to seek solutions to chemical mysteries. To accomplish this goal, an extensive effort has been made to develop experiments that **maximize an inquiry or discovery oriented approach** and **minimize personal hazards and ecological impact.** Simulating a research environment within the constraints imposed by a college course is a demanding challenge. Time limits, safety, chemical toxicity, chemical disposal, chemical costs and equipment costs are a few of the constraints. The preparation of this text has involved a serious effort to overcome these constraints.

In addition to having a positive attitude towards the laboratory portion of the chemistry course, it is very important that the student understands the role of the experience. There is a tendency for students to treat each experiment as a separate unit without sufficient consideration of the connections it makes to other experiments or to the chemical principles being introduced in the lecture portion of the course. During the preparation of this sixth edition, a strong effort has been made to enable students to understand the reason for each experiment's place in the lab and how it adds to the sum of the student's chemical experiences. To help students understand the reasons for doing each experiment and to place the experiments into a broader perspective, this text includes ***Prelaboratory Problems*** with answers to some of the questions, some ***Postlaboratory Problems*** and a set of review exercises in ***Activity 35.*** Most chemistry laboratory courses do not have final exams so the review exercises and ***Postlaboratory Problems*** strive at having the students reflect upon the course and organize the techniques and principles according to applicability and limitations.

A correlation chart between the experiments in this book and concepts in Cengage general chemistry books has been prepared and follows this preface. It should be noted that the sequence of experiments can be changed without an interruption in continuity with a few exceptions. The vanillin prepared in ***Experiment 2*** is used in ***Experiment 3***. The absorption spectrum of triiodide is determined in ***Experiment 25*** and used again in one of the options in ***Experiment 27***. Copper(II) glycinate is synthesized in ***Experiment 28*** and analyzed in ***Experiment 30***.

New features. Because there are many good general chemistry laboratory texts available and an even greater number of in-house texts, this text has made a strong effort to offer many experiments and experiences that are not available in other sources. The use of chromates and lead, barium, mercury and nickel salts has been avoided and less toxic alternatives have been used. Not only does this decrease the exposure of students to hazards, costly disposal costs are minimized. Instead of the commonly encountered titration of chloride with silver, a novel and interesting titration of ferricyanide with zinc is used that includes a determination of the stoichiometry of the reaction. Other unique experiments involving complexes, redox reactions and a study of the triodide - iodine equilibrium have been included because of their educational and technique values. Many instructors do not include organic chemistry in the general chemistry course but most students do not go on to take organic chemistry. Thus it is very important that students gain some experience with distillation, recrystallization and chromatography. Distillation is not commonly included in general chemistry laboratory texts but the distillation of water is included here because of the importance of distillation. As many students have easy access to the Internet, several questions and an improved ***Activity 13*** have been included to familiarize the students with the vast number of resources available at their finger tips. Since

laboratory experimentation is not included in ***Activity 13*** along with ***15*** (molecular models) and ***20*** (organic modeling), the title Experiment has been replaced with the word Activity. ***Activitiy 13*** can certainly be done as an out of class exercise. ***15*** and ***20*** could also be performed outside of class if the students have model kits available. Like many experiments in the text, the molecular modeling experiments include strong efforts to enable the students to gain valuable insight into chemistry. The process of teaching the development of insight deserves more attention in the way chemistry is taught. To improve the amount of insight gained by the students, questions have been added to the Prelaboratory sections and ***Activities 35*** and ***36*** have been reformated or added to this edition. ***Activity 35*** adds many challenges and projects for students that put more of the responsibility on the students. A carbonate analysis that was ***Experiment 24*** in the 5th edition has been removed and inserted into ***Activity 35*** as an independent project for students. ***Activity 36*** is a unique graphing exercise that should develop graphing techniques. Finally, an Internet site developed specifically for use with this text has been recently posted at: http://murov.info/exptsgenchem.htm .

Selection of experiments has been based on a search for strengths such as promoting insight. While two experiments (***9, 26***) introduce approaches to qualitative analysis concepts and techniques, this text does not cover the classical qualitative analysis scheme. The cursory coverage that would have been afforded to the scheme due to page limitations would have been weak. However, we do find that students learn the important concepts of qualitative analysis by performing ***Experiments 9*** and ***26***. Whenever possible, unknowns have been incorporated to promote interest and a research environment. As part of the responsibility of the laboratory portion of the course is to teach technique, most commonly used techniques are studied and applied including organic chemistry techniques such as distillation, recrystallization, and vacuum filtration.

The experiments have been written assuming that a milligram balance will be standard equipment for the course. ***Experiments 2, 3, 4, 12, 14, 17, 18, 19, 24, 28*** and ***33*** can be performed with a centigram balance without significant loss of information. However, use of anything less than a milligram balance for ***Experiments 5, 7, 11, 21, 22, 29, 30*** and ***32*** will result in a decrease in the ability to interpret the results.

This text is designed for the instructor who takes the laboratory portion of the course seriously. Because of the exploratory nature of many of the experiments, students will frequently need guidance. Students may also need to have some of the techniques demonstrated and the discussion extended. Chemistry has been described as "the central science" and the laboratory is the center of chemistry. Students need to approach the laboratory with a positive attitude and be curious, alert observers who are prepared for the unexpected and willing to use their imaginations to seek insights into the mysteries of our universe.

Acknowledgments. I thank my editors, Brendan Killion, Krista Mastroianni, Peter McGahey and Chris Simpson, for giving me the opportunity to further improve this general chemistry laboratory text. My coauthor on another lab text, Brian Stedjee, was the original developer of many of the experiments included here and I am grateful for his willingness to test the experiments, proofread this document and prepare the accompanying instructor's manual. I would also like to thank the many Modesto Junior College students who often had to test primitive forms of the experiments. Many of the images in the 6th edition in the upper right hand corner of each experiment were obtained from clipart that was purchased for the 5th edition from the site: http://www.scs-intl.com/trader/. You might want to challenge yourself to determine the connection between the image and the experiment. My wife, Carolyn Ann Murov, sacrificed valuable time to prepare the figures for this text and for several months approximately every three years has to listen to me cursing at the computer while a new edition is prepared. Finally, I want to thank my four young grandchildren, Dylan (11 in 2013), Hope (11), Carson (9) and Mylee (3) for continually reminding me of the importance of curiosity and the pleasure of finding things out. I sincerely hope students will learn from their chemical laboratory experience and more importantly open their minds to the mysterious and explore it.

Steven L. Murov, Professor Emeritus of Chemistry, Modesto Junior College

Textbook Correlation Table

Murov Expt. #	Key Concepts	Key Techniques	Kotz[1], et. al., Chap.	Masterton[2], et. al., Chap.	Moore[3], et. al., Chap.	Whitten[4], et. al., Chap.	Zumdahl[5] et.al., Chap.
1	scientific method	observation, glassworking	1	1	1	1	1
2	separation of mixtures	evaporation, filtration, recrystallization	1, 13	1,9	1, 11	1, 13	1, 10
3	measurements, sig. figs., density	determination of melting points, densities	1	1	1	1	1
4	accuracy, precision, density	density determination, graphing, pipteting	1	1	1	1	1
5	mole, empirical formulas	pyrolysis, flame tests	2, 3	3	3, 4	2	3
6	classification of chemical reactions, stoichiometry	observation of chemical reactions	3, 4	3, 4	4, 5	3, 6	4
7	stoichiometry	synthesis, filtration	4	3, 4	4	3	4
8	conductivity, electrolytes, bonding	measurement of electrical conductivity	2, 3, 5, 8	4, 7, 9	3, 5	6, 7, 21	2, 4, 8
9	double replacements, net ionic equations	qualitative analysis	3	4	5	6	4
10	single replacements, activities	observational skills	3, 20	4, 17	5, 19	6, 21	4, 18
11	concentrations, stoichiometry	titration	4	3, 4	5, 7	3, 11	4
12	specific heat, enthalpy, Hess's law	calorimetry	5	8	6	15	6
13	periodicity, LD_{50}	navigating Internet	7	6	7	5	7
14	spectroscopy	dilutions, spectroscopy	4, 6	3, 6	7	3, 4	4, 7, App. 3
15	Lewis structures	modeling	8, 9	7	8, 9	7, 8, 9	8, 9
16	polarity, chromatography	chromatography	8	7	9	7	8
17	gas laws	data analysis, graphing	11	5	10	12	5, App. 1
18	phase changes	cooling curves	12, 13	9	11	13	10
19	stoichiometry	distillation, titration	4, 10, 18	1, 4, 14	5, 12	11, 13	4, 10

Murov Expt. #	Key Concepts	Key Techniques	Kotz[1], et. al., Chap.	Masterton[2], et. al., Chap.	Moore[3], et. al., Chap.	Whitten[4], et. al., Chap.	Zumdahl[5], et. al., Chap.
20	organic compounds	modeling	10	22	12	23, 24	22
21	acid and base reactions, stoichiometry	reactions, titration	17, 18	13, 14	16, 17	18, 19	14, 15
22	acid and base stoichiometry	titration	17, 18	13, 14	16, 17	18, 19	14, 15
23	pH, pK_a	pH meter, titration curves	17, 18	13, 14	16, 17	18, 19	14, 15
24	solubility product, free energy	data analysis	18, 19	15, 16	15, 17, 18	17, 20	16, 17
25	equilibrium constant	spectroscopy, data analysis	18, 19	14, 15	14, 18	17, 20	13, 16
26	complex formation	qualitative analysis	18	15	17	20, 25	16
27	kinetics, mechanisms	data analysis, spectroscopy	15	11	13	16	12, App. 3
28	coordination compounds	synthesis	22	19	22	25	21
29	redox, stoichiometry	titration	3, 20	4, 17	5,19	6, 11	4, 18
30	redox, stoichiometry	titration	3, 20	4, 17	5, 19	6, 11	4, 18
31	reduction potentials	observational skills	20	17	19	21	18, App. 5
32	electrochemistry	electrochemical techniques	20	17	19	21	18
33	complex formation	spectroscopy	18	15, 19	17, 22	20, 25	16, App. 3
34	polymers, material selection	observational skills	10	23	11, 12	24, 27	22
35	review, application						
36	graphing		1				App. 1

1. Kotz, J. C.; Treichel, Jr.; P., Townsend, J. *Chemistry and Chemical Reactivity*, 8th ed., Cengage, **2012**.
2. Masterton, W. L.; Hurley, C. N.; Neth, E. *Chemistry: Principles and Reactions*, Cengage, 7th ed., **2012**.
3. Moore, J. W.; Stanitski, C. L.; Jurs, P. C., *Chemistry: The Molecular Science*, 4th ed., Cengage, **2011**.
4. Whitten, K. W.; Davis, R. E.; Peck, M. L., Stanley, G. G., *General Chemistry*, 10th ed., Cengage, **2014**.
5. Zumdahl, S. S.; Zumdahl, S. A. *Chemistry*, 9th ed., Cengage, **2014**.

Chemistry Resources

References can generally be divided into two categories, primary sources and secondary sources. The primary literature references are most commonly the journals that contain the direct reports of the research scientists such as the *Journal of the American Chemical Society*. Searches of information from primary journals usually involves use of an abstracting service such as *Chemical Abstracts*. *Chemical Abstracts* is generally available in university libraries but is very expensive and not usually available in community college libraries. Online access to *Chemical Abstracts* is also expensive. In this course, most of your literature searches will be in secondary sources such as the ***Handbook of Chemistry and Physics*** or the ***Aldrich Chemical Catalog*** and many valuable sites on the Internet. Secondary sources compile data from primary sources into condensed formats and handier tables. In some cases, especially for Internet sites, it is possible that the reference should be considered a tertiary source as the data has been collected from secondary sources. In any event and most importantly, whenever you are writing research articles, most citations should be to primary sources as there are occasional errors made whenever data is compiled.

Compilations of Properties. In some of the experiments you will perform in this course, you might want to look up information on the properties of substances. One starting place is ***Appendix 1*** of this text. College libraries usually will have some of the secondary references listed below. Note that most chemistry books are catalogued in the QD section (or 500 in the Dewey decimal system) of the library although a few are in the physics QC section. It is worth your time to browse the QD part of your library's reference section to give you an idea of which references are available. For each of these references, you will have to learn the fastest way to find data. It is sometimes difficult to figure out what to look for in the index for the ***Handbook of Chemistry and Physics.*** An alternative is to use the ***Aldrich Chemical Catalog***. This commercial catalog includes the physical properties of the organic compounds sold by the company. Ask your instructor if there is an ***Aldrich Chemical Catalog*** available to put in your laboratory. The references listed below are among the best sources of secondary data.

Handbook of Chemistry and Physics, CRC Press, Boca Raton.
Handbook of Chemistry, McGraw-Hill, New York.
Dictionary of Organic Compounds, Chapman and Hall, London.
Merck Index, Merck, N.J.
Aldrich Chemical Catalog, Aldrich, Milwaukee.

While you probably do not own one of the above references, chances are that you have access to a computer with online access. The Internet has many sites that contain physical properties of substances. Some present the data directly when you input the name of the compound. In addition, many sites contain hotlinks to MSDS (Material Safety Data Sheet). The MSDS usually contains property data in the 9th section on physical properties. For toxicity data, the Chemidplus site is often the most convenient resource.

Direct links to the sites in the lists on the next page have been included in a site that has been specially prepared for use with this text. In addition, many other useful sites are also included. It is recommended that you visit this site and notice what is available as this site might save you substantial time in your searches for information. The first section of the site contains suggested links arranged by experiment number. The most useful sites for locating properties are included in a section on the site as well as on the next page.

Experiments in General Chemistry web site:

http://murov.info/exptsgenchem.htm

1.Tabulation + MSDS

Acros - http://www.acros.com/portal/alias__Rainbow/lang__en/tabID__21/DesktopDefault.aspx
Aldrich, Sigma - http://www.sigmaaldrich.com/
Alpha Chemical - http://www.alfa.com/alf/laboratory_chemical_suppliers.htm
ChemicalBook - http://www.chemicalbook.com/
ChemExper Chem. Directory - http://www.chemexper.com/
ChemFinder Web Server - http://chemfinder.cambridgesoft.com/
Chemidplus - http://chem.sis.nlm.nih.gov/chemidplus/
or http://chem.sis.nlm.nih.gov/chemidplus/chemidlite.jsp
Chemspider - http://www.chemspider.com/
EMD Chemicals - http://www.emdchemicals.com/corporate/emd_corporate.asp
Fisher - https://new.fishersci.com/wps/portal/HOME
TCI America - http://www.tciamerica.com/brochure/catalog.html
Wikipedia - http://en.wikipedia.org/wiki/List_of_organic_compounds
http://en.wikipedia.org/wiki/List_of_inorganic_compounds

2. MSDS

JT Baker & Mallinckrodt (primarily inorganic chemicals) -
http://www.mallbaker.com/Americas/catalog/default.asp
MSDSprovider - http://www.msdsprovider.com/
MSDS Solutions - http://www.msds.com/
Vermont Safety Information Resources, Inc. - http://hazard.com/msds/

3. Tabulation

Chemblink - http://www.chemblink.com/
Chemfate - http://www.syrres.com/esc/chemfate.htm
Chemical Database - http://ull.chemistry.uakron.edu/erd/
Chemland 21 - http://www.chemicalland21.com/listaz01.htm
CIS - http://www.ilo.org/legacy/english/protection/safework/cis/products/icsc/dtasht/index.htm
Datalog - http://www.syrres.com/esc/datalog.htm
Knovel Critical Tables (free registration required) -
http://www.knovel.com/web/portal/browse/display?_EXT_KNOVEL_DISPLAY_bookid=761
Select the Table for *Basic Physical Properties of Chemical Compounds* and register.
Use the sort by ascending or descending order capability for any parameter and also the filter capability.
LookChem - http://www.lookchem.com/
National Physical Laboratory - http://www.kayelaby.npl.co.uk/chemistry/3_3/3_3.html
WolframAlpha - http://www.wolframalpha.com/

Chemistry experiments employing the use of chemicals and laboratory equipment and 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 has provided, for your convenience only, safety information intended to serve as a starting point for good practices. Cengage Learning makes no guarantee or representations as to the accuracy or sufficiency of such information and/or instructions.

SAFETY FIRST

and Prudent Laboratory Practice

While many people think that the chemical industry must rank high on a list of frequency of accidents, the reverse is actually true. The days lost per person due to accidents in the chemical industry is one of the lowest for all professions. This is not because of fewer potential hazards. To the contrary, the chemical laboratory is full of accidents waiting to happen. However, proper precautions and patience prevent the accidents from happening.

1. Come to the laboratory prepared. Read the experiment and study it. Think about manipulations where special care is needed.

2. Wear eye protection at all times. Your eyes are a very valuable part of your body but are also one of the most vulnerable parts. And don't forget, you are not the only student in the lab. Sometimes chemical accidents spread chemicals throughout the lab.

3. Do not perform unauthorized experiments. Sometimes there is a strong temptation to just mix chemicals together. Do not succumb to this temptation. On the other hand, exploration is strongly encouraged. If you want to try your own experiment, first try to predict its outcome. Then discuss it with the instructor and obtain his/her approval and guidance. Finally for previously untested reactions, use very small quantities and run the reaction in the hood behind a safety shield.

4. When you first enter the laboratory, memorize the locations of the fire extinguishers, eye wash, safety shower, first aid kit and exits from the laboratory. The instructor should discuss the use of each type of safety equipment. If, despite your eye protection, something should get into your eye, thoroughly wash your eyes for several minutes with the eye wash. Don't stop after a brief rinsing.

5. Conduct all experiments that evolve gases or unpleasant odors in the hood and make sure the hood is on and functioning.

6. Never take food or drinks into the laboratory.

7. Do inform the instructor of all accidents, even the most minor of cuts or burns.

8. Pay special attention to the names and concentrations of chemicals and read bottle labels carefully. If an experiment calls for addition of hydrogen peroxide to bleach to generate oxygen and hydrochloric acid is added instead, lethal chlorine gas will be generated that can endanger the lives of the whole class.

9. Treat glass tubing with great respect. Chemical accidents such as the one described in #8 above fortunately seldom occur but minor accidents with glass tubing occur too frequently. In ***Experiment 1***, be very patient and let the hot glass cool before you touch it. Firepolish the ends of all pieces of glass tubing to avoid cuts. Most important, when inserting a piece of glass tubing through a hole in a rubber stopper:

    a. lubricate the hole or glass tubing
    b. hold the tubing very close (within 1 cm) to the part of the tubing that is about to enter the stopper
    c. wrap the tubing in a cloth towel
    d. push slowly while slowly rotating and be patient.

10. If you touch a lab reagent bottle and find that it is wet, put it down and wash your hands.

11. Be sure whenever you are working in the lab that another person familiar with lab safety guidelines is present and that the instructor is within shouting distance.

12. Wear appropriate clothing that covers most of your body. A lab apron is also helpful. Do not wear floppy, loose clothing such as neckties or scarves. Do not wear sandals or open toed shoes.

13. Do not ever leave a heated experiment unattended and do not ever heat a closed system such as a stoppered flask or test tube. Never heat very volatile liquids such as ether or pentane with a flame and in fact don't even use them in rooms with flames burning.

14. Keep your section of the lab clean and treat all equipment, especially the balance as though it is part of your very own Porsche.

15. Carefully clean up all spills and breakages. If you break a mercury thermometer or spill a corrosive chemical, inform your instructor immediately.

16. Keep your books away from sinks especially those that are equipped with aspirators. Occasionally drain hoses jump out of sinks and drench everything near the sink.

17. Do not put chemicals into the sink without first obtaining the instructor's permission.

18. When taking a chemical from a bottle, try not to take more than you need. If you exceed your need, do not return it to the bottle. Give it to another student or properly dispose of it.

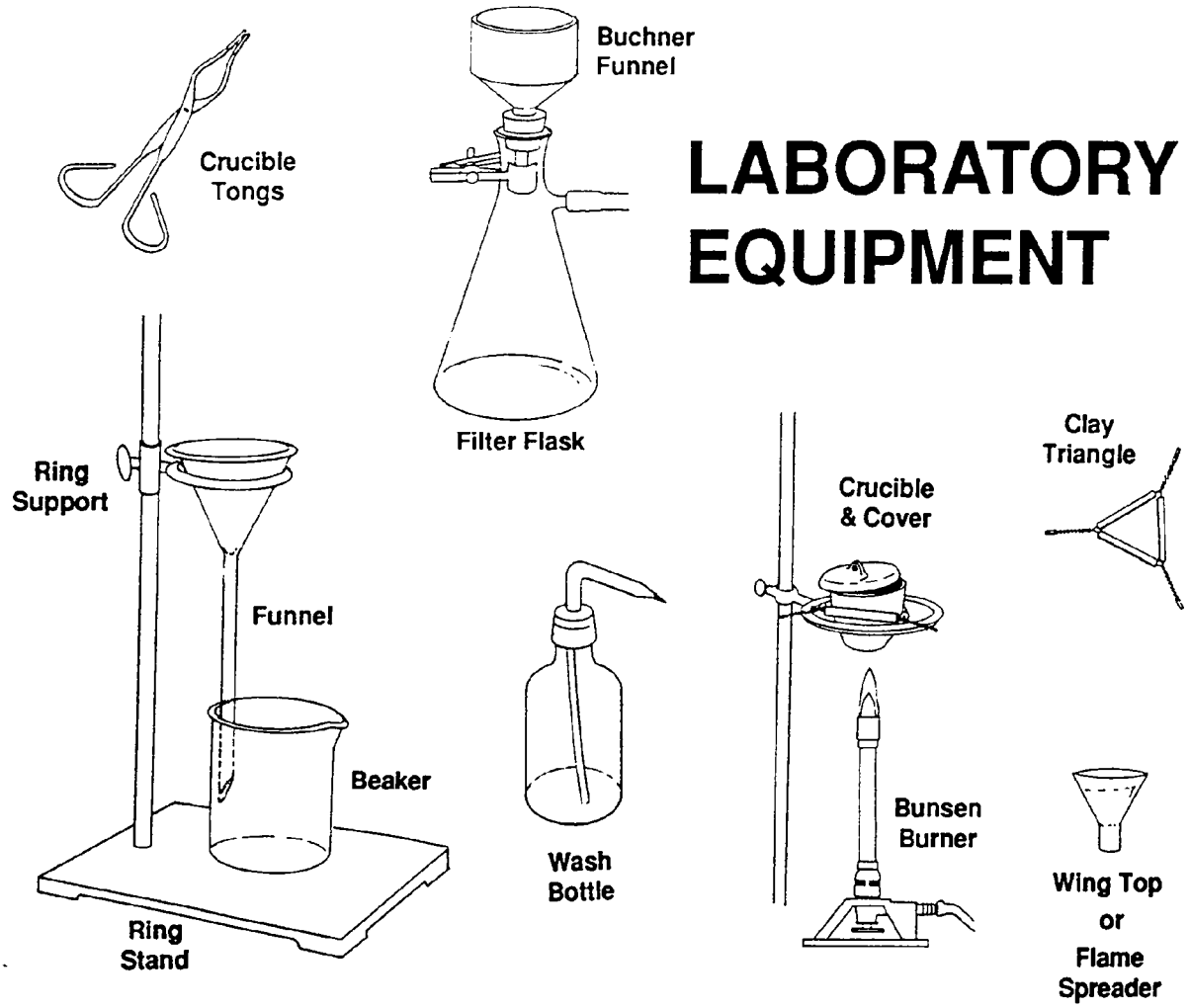
19. Do not put spatulas, stirring rods, droppers or pipets into chemical bottles. Transfer the chemical to your own container first. If the chemical will not pour out, seek help from the instructor.

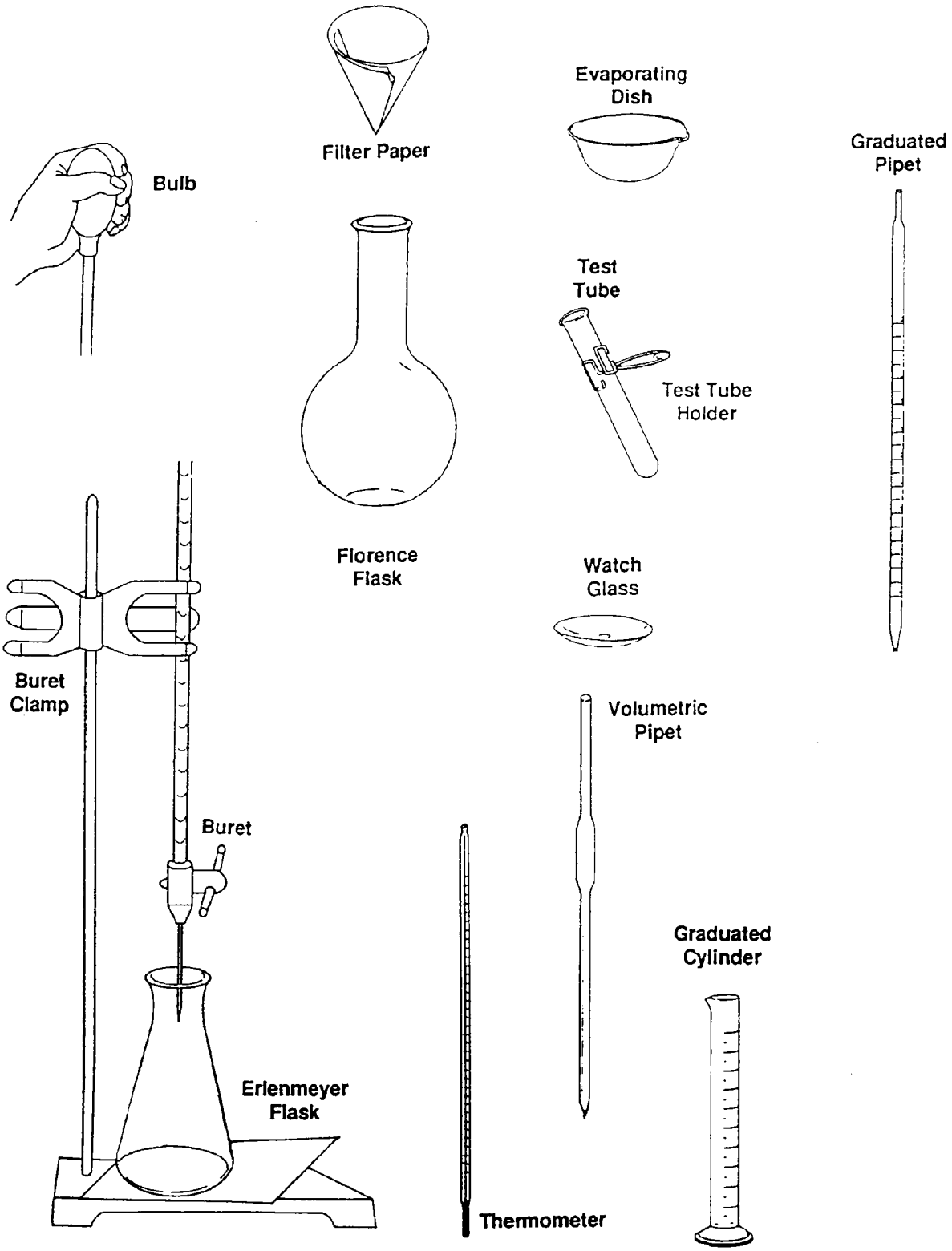
20. Do not put bottle stoppers down on the desk top. This could contaminate the contents of the bottle.

21. If, when using a reagent bottle, you get the bottle wet, rinse off its outside and dry the bottle.

22. Remember that all chemicals have some degree of toxicity. Every effort has been made in the design of this lab text to use chemicals that minimize toxicity and disposal problems. Recognize, however, that the chemicals included here are toxic and need to be treated with care and respect.

23. Do wear eye protection, think, be patient, explore, learn, enjoy and show your enthusiasm.





Experiment 1

EARLY EXPLORATIONS AND TERMINOLOGY

1865 Bunsen burner

Learning Objectives

Upon completion of this experiment, students will have experienced:
1. Practice with techniques of observation and the scientific method.
2. The use of the Bunsen burner.
3. Basic glassworking.
4. Some basic chemistry terminology.

Text Topics

Scientific method, physical properties, chemical reactions (for correlation to some textbooks, see page ix).

Notes to Students and Instructor

This experiment will probably take more than one laboratory period. It can be started on the first lab day which usually includes check-in procedures and a safety presentation and then finished on the second lab day. Alternatively, selection of only certain sections should shorten the experiment. ***Most sections of this experiment should be thoroughly discussed in class after completion of the experiment.***

Discussion

Pretend for a moment that it is a very hot day. Use your imagination to visualize a glass of ice water. Have you noticed that the ice is floating in the water and not resting on the bottom of the glass? Has this observation ever puzzled you? ***Observation and imagination are two of the keys to good science.*** Mysteries that arise from inconsistencies between expectations and observations often contain clues that lead to exciting and wonderful discoveries.

Consider the ice-water system. What should happen to the density (mass/volume) of a liquid as it is cooled? We might expect a contraction of volume and an increase in density as the temperature decreases (resulting in sinking). And yet, contrary to this expectation, the ice is less dense than liquid water and floats. Included in the observation of the ice-water system then should be the unexpected floating of the ice and questions about why the ice floats and if it is common for the solid phase of a substance to float in its liquid.

Learn to make careful, complete and unbiased observations and include as part of these observations, questions on any inconsistencies that arise from them. Ideally observations should not depend on the observer as we are trying to **record facts in an understandable way for other people**. It is important that scientific observations be reproducible. The observation section of a report should not include interpretations or explanations because explanations might differ from one observer to the next and there may even be more than one possible conclusion. Ice sometimes forms with small air bubbles and in these cases one should record that there are air bubbles in the ice. But to say that the ice floats because of air bubbles is not an observation but in this case an inadequate explanation.

It is very important to record all observations as the act of disregarding or ignoring is actually a conclusion that an observation is not important. Some very important observations have been overlooked only to be found by later investigators to have significance (penicillin and nuclear fission are two examples). Discoveries of teflon and aspartame were made serendipitously by careful observers who did not overlook the unexpected. When doing science, pay heed to the words of Ralph Waldo Emerson, ***"God hides things by putting them near us.,"*** and Louis Pasteur, ***"In the fields of observation, chance favors only the mind that is prepared."***

Observation is the first part of a process commonly called ***the scientific method***. Although its emphasis in some textbooks sometimes gives the misleading impression that scientists operate according to a schedule, the scientific method does describe the process that occurs in scientific exploration. It starts with the puzzling observation and resulting questions. Next with the use of imagination, explanations (or hypotheses) are suggested. Fortunately in science (and this is what makes science easier than most fields), explanations are testable. If experiments support an explanation, the explanation becomes a theory. The theory is always subject to further testing which can result in modification or even discarding of the theory.

As you do your laboratory experiments, remember to stay alert and record all observations including questions about anything curious to you. Be sure that your records are written clearly and concisely in a way that can be understood and tested by others.

Procedure

This exercise has been designed to help you develop your observational skills, distinguish between observations and explanations, and to learn to carefully record all observations. Remember that complete observations often lead to questions.

A. The meniscus. Add water to about the half way point of a 50 mL graduated cylinder and study the features of the water surface. Describe and draw your observations. The phenomenon that you observe is called a meniscus. Write down a question about the meniscus. [Comment: Volumetric glassware has been calibrated to give correct volume measurements when you read the **very** bottom of the meniscus.]

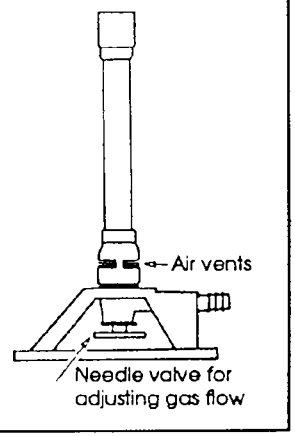
B. The candle flame. Light a candle and study the flame. Write down all your observations and questions about the flame. Try to include observations on states of matter and physical and chemical properties and changes. Be very careful to distinguish observations from explanations.

What do you think is actually burning? Write your answer down before you read or experiment further. You will not lose points for an incorrect answer.

Be sure to record observations from the following tests. Put a beaker over the burning candle almost but not all the way down and carefully observe the inside of the beaker. Blow the candle out and immediately put a glass stirring rod on top of the extinguished wick in the region where the flame had been. Inspect the rod. Relight the candle, and, with a burning match in one hand, again blow the candle out. Immediately bring the burning match to the region where the flame had been, moving the match slowly towards the wick for the last 2 cm. Save the candle for Part L.

C. The Bunsen burner. One of the important tools of the laboratory chemist is the Bunsen burner. This exercise has been designed to familiarize you with the burner and introduce you to glassworking. Study *Figure 1-1* and compare it to your Bunsen burner.

Fig. 1-1



[Comment: The instructor should demonstrate use of the burner.] Close the air control and make sure the gas valve is off. Turn the gas valve on the bench on full, light a match, open the burner gas valve and light the gas. What color is it? Increase the gas flow until the flame is about 4 cm. high and open the air control until the yellow color is gone. Draw and describe the flame. What do you think is the hottest region? Take a wood splint and insert it quickly into the flame right over the top of the burner (*Figure 1-2*). Hold it there until it ignites and observe where it burns. Now hold a wire gauze vertically in the flame so that about 1 cm. of the gauze extends beyond the far edge of the burner (*Figure 1-3*). Heat it until it glows and record your observations about the position and pattern of glow. If significant amounts of colored flame leap from the gauze, rinse the corner of the gauze that you are heating with deionized water and repeat the test.

Fig. 1-2

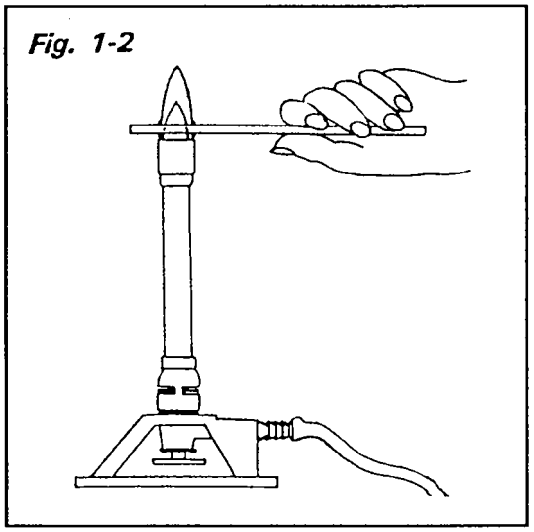
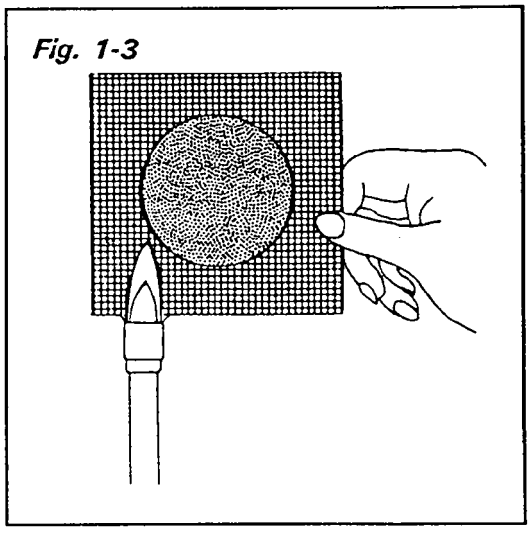
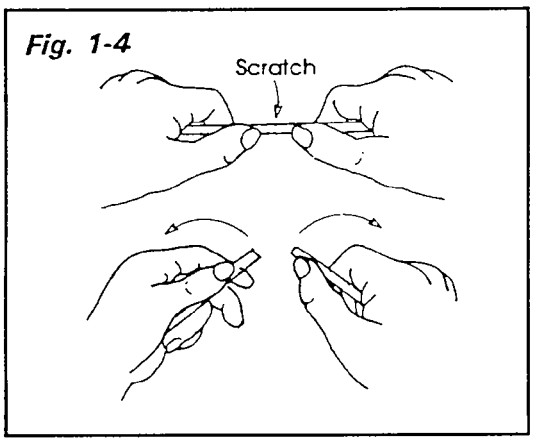
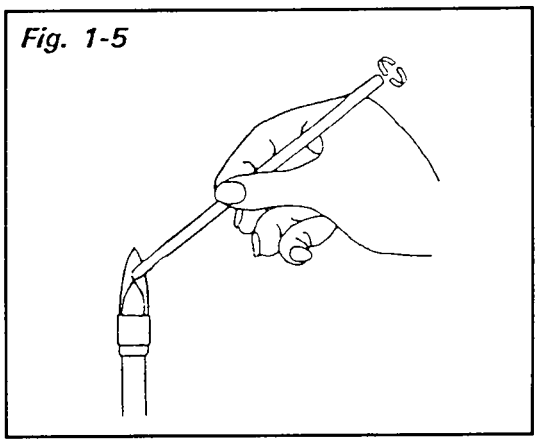


Fig. 1-3



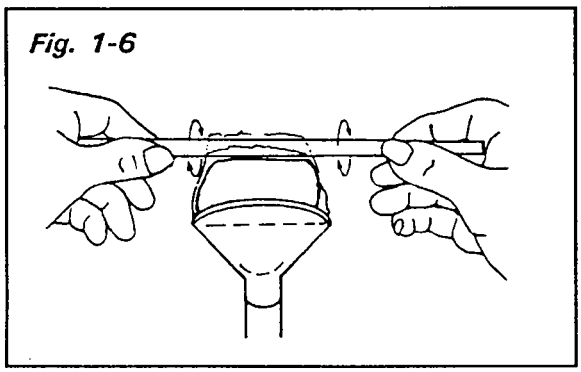
Glassworking. Take a file and make a deep scratch on a piece of 6 mm glass tubing 20 cm from the end. Be sure not to make more than one groove. Holding the scratch away from you with thumbs on either side of the scratch, push your thumbs forward, pulling the two pieces apart (*Figure 1-4*). With a decent scratch, the glass will almost split by itself. However, the ends will still be sharp enough to cut you. Anytime glass tubing is cut, the ends should be firepolished to round off the sharp edges.

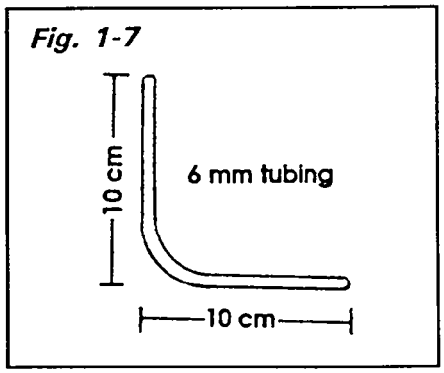




Firepolishing. Following *Figure 1-5*, hold the tubing at about 30° to the horizontal in the hottest part of the flame. Rotate the tubing and observe it carefully. As it approaches its melting point, a bright sodium flame will be observed. Continue to rotate it until it barely melts. Too much melting will begin to constrict the tube opening. Put the tubing down on a wire gauze until it has cooled and firepolish the other end (***Caution: One of the most common lab accidents is the burning of fingers and hands on glass that has been picked up without sufficient cooling.***)

Bending tubing. Chemists often have to make their own specialized pieces of glassware and it is very useful to have some experience with bending glass. Turn off the Bunsen burner and insert the flame spreader (often called a wing tip). Relight just as you did without the flame spreader and adjust the flame as in *Figure 1-6*. Hold both ends of the tube, place it in the hot region of the flame and rotate rapidly. A relatively even yellow glow indicates even heating. After the tube has softened, remove it from the flame, bend it to a right angle and hold it steady for a few seconds. Place it on the gauze for cooling. Do not be disappointed if your bend is not too aesthetically appealing as good glassworking takes many hours of practice.





D. Rate of mixing. Mount a 250 mL beaker half full of water on a wire gauze above a Bunsen burner. Heat the water to boiling. Using beaker tongs, pour 50 mL of the hot water into a 150 mL beaker. Add 50 mL of room temperature water to a second 150 mL beaker. Use a dropper bottle of food coloring to add a drop of food coloring to each beaker. Hold the dropper over the lip of each beaker, squeeze out one drop and allow it to fall into the water. Try to add the drop to each beaker in the same way. Do not disturb the beaker in any way but carefully observe the beakers for several minutes and then look at them again in about a half hour.

E. Tearing paper. Try to tear a piece of paper (newspaper is preferable) first vertically and then horizontally. Attempt to make the tears fairly rapidly as opposed to doing it in very small sections with your fingers restricting the direction. Describe and explain your observations.

F. Chemistry references. See ***Preface*** pages xi - xii. It is very important to be familiar with the resources that are available to find information about the properties of substances. Hardcover books such as the *Handbook of Chemisry and Physics* and the *Aldrich Catalog* are very valuable sources of chemical properties. Today, Internet accessibility provides a huge library of information and experience with the various sites can make the search much easier and faster. Links to many valuable sites are available at: http://murov.info/exptsgenchem.htm. Included is a link to the very useful source: http://chem.sis.nlm.nih.gov/chemidplus/chemidlite.jsp. Go to this site and enter acetic acid into the search box. Under physical properties find the formula, melting and boiling points of acetic acid and under toxicity, find the human TD_{Lo} value and the rat LD_{50} value. Use the Internet to find the meaning of the notations, TD_{Lo} and LD_{50}. Also find at least one household product that contains acetic acid.

G. Terminology. Terminology and nomenclature are extremely important in chemistry. An understanding of the language of chemistry makes it much easier to communicate with other chemists and to understand their observations and results. In fact without a working ability with chemical terminology and nomenclature, the transfer of knowledge is close to impossible. This exercise will provide an experience with some of the descriptive terms used routinely by chemists. Early in your chemistry textbook and perhaps in the glossary there should be a discussion of each of the terms used here and in the Prelaboratory problem number 7. For this exercise, find at least one example of each term in your laboratory and describe the example. If appropriate, give its use and location. It is permissible to use observations of the exercises in other parts of this experiment as examples.

H. Solutions. In this procedure, you will apply terminology to some observations on solutions of sodium tetraborate and on the temperature dependence of the solubility of this compound. Add about 200 mL of water to a 400 mL beaker. Place the beaker on a hot plate or on a wire gauze supported above a Bunsen burner and heat the water to boiling. *[Caution: Always use beaker tongs when manipulating beakers of hot liquids. Do not use crucible tongs for beakers.]* While waiting for the water to boil, add about 0.05 gram of sodium tetraborate [$Na_2B_4O_7 \cdot 10\,H_2O$] to about 5 mL of water in a 18x150 mm test tube. Mix the contents of the tube by firmly grasping the test tube between your thumb and forefinger of one hand and striking the bottom of the test tube vigorously and frequently with the forefinger of your other hand. ***Never*** put a thumb over the mouth of the test tube to avoid spilling when shaking. If the method above does not achieve adequate mixing, insert a cork or rubber stopper into the tube and shake as you hold the stopper down with

your thumb. Continue mixing until changes are no longer observable. Does the $Na_2B_4O_7 \cdot 10\,H_2O$ completely dissolve and what terminology can now be applied to the mixture? Add an additional 0.05 g of $Na_2B_4O_7 \cdot 10\,H_2O$ and repeat the mixing and observing process. Now add an additional 2 grams of $Na_2B_4O_7 \cdot 10\,H_2O$ to the solution and attempt to repeat the dissolving process. If it doesn't dissolve, put the test tube in the beaker of boiling water and stir the mixture in the test tube with a stirring rod until the sodium tetraborate dissolves. Place the test tube in a rack or beaker and allow it to cool to room temperature. After several minutes, report your observations or, if nothing happens, scratch the inside of the tube with a glass rod and let it sit for several minutes and then report your observations.

I. Colors. When an atom loses or gains electrons it becomes an ion. Positive ions are called cations and negative ions are called anions. In this experiment, you will study dilute solutions of compounds which have ionic bonds. This means that when the compounds dissolve in water, dissociation into cations and anions occurs. Ions can be colored or colorless and generally the color of an ion does not depend on its partner [sodium chloride and sodium nitrate solutions are colorless but copper(II) sulfate and copper(II) nitrate solutions are blue because sodium, sulfate, and nitrate ions are colorless and copper(II) is a blue ion]. If we had solutions available for you containing all of the commonly encountered ions, you would notice that most of the common ions are colorless. ***This makes it very useful for identification purposes for you to observe and remember the names and colors of the colored ions.*** Often you can suspect upon visual inspection that an unknown blue solution contains copper(II) ions.

Two sets of samples will be provided in sealed vials. Each sample will be an ionic compound dissolved in water with the concentrations all about the same (0.1 M). For the first set, you should focus your attention on the colors of the cations. Assume that the anions (either chloride, nitrate or sulfate) are colorless and look for a correlation between color and position in the periodic chart. For the second set, focus your attention on the colors of the anions (sodium and potassium ions are coloress).

Set 1:	aluminum nitrate	iron(III) chloride	potassium chloride
	barium chloride	lead(II) nitrate	silver nitrate
	calcium chloride	lithium chloride	sodium chloride
	cerium(III) nitrate	magnesium chloride	strontium chloride
	cerium(IV) sulfate	manganese(II) chloride	tin(II) chloride
	chromium(III) chloride	mercury(I) nitrate	tin(IV) chloride
	cobalt(II) chloride	mercury(II) nitrate	zinc nitrate
	copper(II) chloride	nickel(II) chloride	
Set 2:	sodium acetate	potassium ferricyanide	potassium permanganate
	sodium bromide	potassium ferrocyanide	sodium phosphate
	sodium carbonate	sodium hydroxide	sodium sulfate
	sodium chlorate	sodium iodate	sodium sulfite
	sodium chloride	sodium iodide	sodium thiocyanate
	sodium chromate	sodium nitrate	sodium thiosulfate
	sodium dichromate	sodium oxalate	

J. Chemical reactions. Upon mixing two solutions, the four common observations that indicate that a chemical change has occurred are: formation of an insoluble product (precipitate), bubbles (or evolution of a gas), heat, a color change. The absence of these observations often but not always means that there has not been a chemical reaction as a result of the mixing. Negative results are very common and just as important as positive results and must be appropriately recorded. You will prepare 5 different mixtures, make observations and determine if a reaction has occurred. Pour **about** 2 mL of the first solution into a test tube and add about 2 mL of the second solution, mix and observe. Notice that there are times when accuracy and precision are extremely important in chemistry and other situations where the results do not change over a wide range of amounts. For the latter cases, it is a waste of time to spend long amounts of time carefully measuring the amounts. These reactions are examples of cases where 2 mL means between about 1 mL and 3 mL and therefore can be estimated. It might be wise to measure two mL once with a 10 mL graduated cylinder and transfer it to a test tube to give you an idea of the volume.

System	Solution 1	Solution 2
A	calcium chloride (0.1 M)	sodium carbonate (0.1 M)
B	hydrochloric acid (3 M)	sodium hydroxide (3 M)
C	calcium chloride (0.1 M)	potassium nitrate (0.1 M)
D	sodium carbonate (1 M)	hydrochloric acid (3 M)
E	iron(III) chloride (0.1 M)	potassium thiocyanate (0.1 M)

K. Mystery flask. Add the solutions below to a 125 or 250 mL Erlenmeyer flask, swirl until mixed and allow to stand without agitation until a change has been observed.

15 mL of a dextrose (glucose) solution (80 g/L)
15 mL of a potassium hydroxide solution (64 g/L)
10 drops of a methylene blue solution (0.4 g/L)

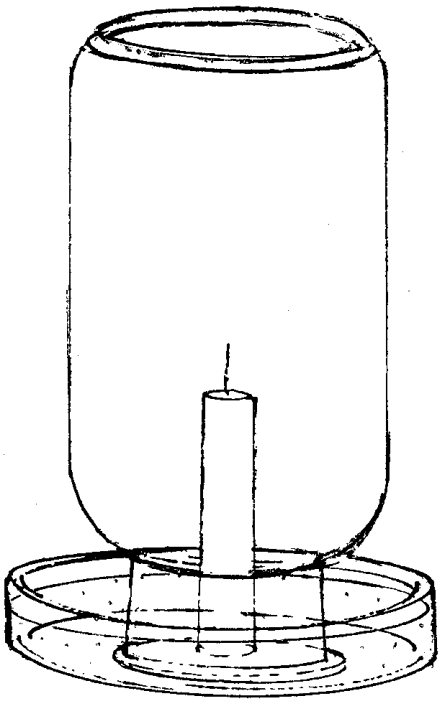
Record all of your significant observations. Vigorously swirl the solution for several seconds and again record your observations. Repeat the sequence as often as you desire but focus your attention on the change that occurs when you swirl the mixture. Save this solution in case you decide later that you want to experiment further with it.

L. Classic Burning Candle Experiment. Light a candle that is at least 6 cm long, drip some melted wax into the middle of a 100 mm x 20 mm Petri dish and set the candle into the melted wax (*Figure 1-8*). After the wax solidifies, fill the Petri dish with water and ignite the candle. Place an inverted gas collecting bottle over the candle and stand it in the Petri dish. Observe the flame and the water level as soon as the bottle has been put in place. Measure the height of the water in the bottle after no more change is apparent. Also devise a method to determine the percentage of the volume filled by the water and perform the measurement.

One of the most important criteria for the testing of a scientific hypothesis is **reproducibility**. One hypothesis that was suggested many years ago to explain the observations in this experiment is that the burning of the candle uses up the oxygen in the bottle and the water rises to replace the used up oxygen. Repeat the experiment a number of times until you are confident the

results are either reproducible within experimental error or not reproducible within experimental error. In the event that you come to the latter conclusion, try to come up with a new hypothesis to explain your observations. If possible, devise experiments to test your new hypothesis and with the permission of the instructor, perform the experiments.

Fig. 1-8



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Some quotations related to the importance of making careful, complete and unbiased observations.

Shakespeare says, we are creatures that look before and after: the more surprising that we do not look round a little, and see what is passing under our very eyes. Thomas Carlyle

You see but you do not observe. (Sherlock Holmes to Dr. Watson) A. Conan Doyle

Observation, not old age, brings wisdom. Publilius Syrus

Armado: How has thou purchased this experience?
Moth: By my penny of observation. William Shakespeare

In the fields of observation, chance favors only the mind that is prepared. **Louis Pasteur**

God hides things by putting them near us. **Ralph Waldo Emerson**

Name_____Date_____Lab Section_____

Prelaboratory Problems - *Experiment 1* - Early Explorations and Terminology

The solutions to the starred problems are in ***Appendix 4***.

1. What color is water? _____

Problems 2 - 5 describe some observations you have probably made. But have any of the observations stimulated you to the point where you asked a question about them? Try to come up with a question now and suggest an explanation.

2. Popcorn pops when heated sufficiently.

3.* Vinegar and oil do not mix.

4. Your laboratory drawer has Erlenmeyer flasks and beakers that hold similar volumes.

5.* The necks of volumetric flasks and pipets have very small diameters.

6. Give observations for *Figure 1-9*.

Fig. 1-9

7. Classify the following items a - r using the number codes for the terms below. In some cases, more than one term might be applicable.

1 substance	4 homogeneous mixture	8 intensive physical property
2 element	5 heterogeneous mixture	9 extensive physical property
3 compound	6 saturated solution	10 physical change
	7 unsaturated solution	11 chemical change

a.* gold _____	g. density _____	m. rusting _____
b.* vinegar _____	h. water _____	n. iodine _____
c.* vinegar & oil _____	i. sodium chloride _____	o. orange juice _____
d.* volume _____	j. evaporation _____	p. vodka. _____
e.* melting point _____	k. smog _____	q. coal burning _____
f.* dilute salt water _____	l. freezing of water _____	r. carbonated soda _____

8. Compounds are composed of two or more elements. Are the properties of compounds something like an average of the properties of its component elements? Consider for example sodium chloride (NaCl) and iron(III) oxide [commonly called rust (Fe_2O_3)]. Explain your answer.

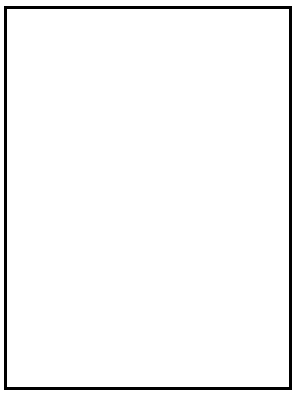
Name_____Date_____Lab Section_____

Results and Conclusions - *Experiment 1* - Early Explorations and Terminology

A. The meniscus.

1. Description:

2. Drawing



3. Question about the meniscus:

B. The candle flame.

1. Observations (include comments on states of matter, physical and chemical changes):

2. What do you think is sustaining the flame (e.g., burning of the wick, the solid wax, the liquid wax or wax vapor)? Explain your answer.

3. Further observations:

   a. beaker partially over burning candle -

   b. stirring rod next to extinguished wick -

   c. burning match approaching extinguished wick -

4. Was your first explanation (question 2) correct or do you want to modify it or suggest a new one? Explain your answer.

C. The Bunsen burner.

1. Draw a picture of the flame. Based on your results with the splint and the gauze, indicate the hottest and coolest regions of the flame.

D. Rate of mixing.

1. Initial observations:

   a. room temperature water

   b. hot water

2. Observations about 0.5 hour later:

   a. room temperature water

   b. hot water

3. When two aqueous solutions are introduced into the same container, is stirring needed to achieve a homogeneous system? Explain your answer.

4. How would you stir a solution freshly prepared in a volumetric flask?

E. Tearing paper

1. Describe your observations when you attempted to tear the paper vertically.

2. Describe your observations when you attempted to tear the paper horizontally.

3. Suggest an explanation for any differences observed between vertical and horizontal tearing.

F. Chemistry references. acetic acid formula _____

b.p. _____ m.p. _____

TD_{Lo} _____ LD_{50} _____

Meaning of TD_{Lo} _____

Meaning of LD_{50} _____

Household product(s) that contain acetic acid _____

G. Terminology (Describe at least one example of each of the following in your laboratory.)

1. substance

2. element

3. compound

4. homogeneous mixture

5. heterogeneous mixture

6. saturated solution

7. unsaturated solution

8. chemical change

9. Shortly after the Chernobyl nuclear reactor accident, some people took potassium iodide tablets to dilute the radioactive iodide in their bodies and diminish its retention in the thyroid gland. Although many experts questioned this practice, little harm was probably caused because of the relatively low toxicity of KI. However, on May 12, 1986, *Newsweek* incorrectly captioned a photo that showed a child apparently receiving KI with "On Alert: Administering iodine to Polish children." Critically evaluate the mistake and possible consequences.

H. Solutions. [Be sure to use appropriate terminology from the section above for all of these questions.]

1. Describe the test tube contents after you have added 0.05 gram of sodium tetraborate decahydrate and stirred.

2. Describe any significant changes after an additional 0.05 gram of $Na_2B_4O_7 \cdot 10\,H_2O$ is added and mixed.

3. How can the sodium tetraborate - water system be distinguished from a compound?

4. Describe your observations when an additional 2 grams of $Na_2B_4O_7 \cdot 10H_2O$ is added to the solution.

5. What happens to the mixture in *#4* when it is heated?

6. Describe what happens when the solution from *#5* is allowed to cool. Are your observations consistent with your expectations? Explain your answer.

I. Colors.

1. List the colored cations and their colors.

cation	color	cation	color	cation	color
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____

2. Can you make any generalizations about color versus position in the periodic chart?

3. List the colored anions and their colors.

anion	color	anion	color	anion	color
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____		

J. Chemical Reactions.

Reaction	Observations
A	
B	
C	
D	
E	

K. Mystery flask.

1. Observations upon standing:

2. Observations upon swirling:

3. Suggest an explanation for the change that occurs upon swirling.

4. How could you test your explanation (it might be possible with your instructor's approval to actually perform the test).

L. Classic Burning Candle Experiment.

1. Trial 1 water level (distance above dish water level) _____
2. Volume of water that moved into bottle _____
3. Volume of bottle _____
4. Percentage of bottle filled by water _____
5. Additional trial water levels _____ _____ _____ _____
6. Based on the percentage of the bottle filled by the water, is it likely that the water simply displaced used up oxygen? Explain your answer.

7. Based on the reproducibility of the results, does the hypothesis that the water is displacing used oxygen fit the observations? Explain your answer

8. If your answer to number 7 was negative, suggest another hypothesis to explain your observations.

9. Suggest and if possible perform experiments to test your hypothesis in number 8. Describe the experiments and their results. Do the experiments support or refute your new hypothesis?

10. Some of the *Learning Objectives* of this experiment are listed on the first page of this experiment. Did you achieve the *Learning Objectives*? Explain your answer.

Experiment 2

SEPARATION OF MIXTURES


early filtration apparatus

Learning Objectives

Upon completion of this experiment, students will have experienced:
1. The evaporation of water from salt water.
2. The separation of a solid from a liquid using gravity filtration.
3. Recrystallization and use of vacuum filtration.

Text Topics

Physical properties, separations of mixtures using physical properties, temperature effects on solubility (for correlation to some textbooks, see page ix).

Notes to Students and Instructor

The vanillin recrystallized in this experiment will be saved for ***Experiment 3*** where its percent recovery and purity will be determined.

Discussion

Imagine yourself by a river in the mountains. You are thirsty but is it safe to drink the water? Probably not because the river is not just water but is a mixture of several substances and probably contains bacteria that can cause intestinal problems. Almost everything that we encounter in nature is a mixture and this adds a challenge to the work of chemists. Analytical chemists may need to know what substances are present (e.g., are pesticides in a water supply?) and this usually involves separation and purification before some type of identification test can be performed. Most physical and chemical tests on mixtures do not give results that can be easily interpreted. The chemist may also need a substance for use as a starting material in a chemical reaction. The presence of impurities may affect or even eliminate the desired reaction. Even chemicals obtained from the stockroom are not absolutely pure with grades of purity generally ranging between 90 and 99.99%. Sometimes small amounts of impurities can be tolerated and in other cases, they must be removed. Would you want as much as 0.1% of a toxic substance in a medicine?

Many separation and purification techniques have been developed. You will use several during this course and three important ones in today's experiment. Try to consider the applicability of each technique as you use it. Can it be used on large samples or only small ones? Will it work to remove large amounts of impurities or only trace constituents? Will it work on any of the three common states of matter or only one? The three techniques you will use today all take advantage

of differences in physical properties of the substances being separated. The evaporation of water from salt water to leave behind the desired sodium chloride utilizes the differences in boiling points of water and sodium chloride. The gravity filtration to collect calcium carbonate takes advantage of the very low solubility of calcium carbonate in water and the ability of filter paper to allow passage of a liquid but not a solid. Finally the recrystallization of vanillin relies on the observation that solubility of a solid in a liquid often increases with increasing temperature.

Procedure

A. Evaporation: Determination of the mass percent of sodium chloride in a saturated sodium chloride solution. The amount of sodium chloride dissolved in a saturated solution can be determined by evaporating a weighed amount of a saturated solution to dryness. Dryness is confirmed by repeated heating and weighing cycles until a constant mass is achieved. Decant (be careful not to agitate) about 6 mL of a saturated sodium chloride solution into a 10 mL graduated cylinder. Weigh a clean, dry, evaporating dish to the nearest 0.01 g (preferably 0.001 g) and add the 6 mL of NaCl solution to it. Reweigh the dish and its contents.

Put a 400 mL beaker about half full of water on a wire gauze above a Bunsen burner. Suspend the evaporating dish in the 400 mL beaker. Boil the water (add water as needed to the beaker to maintain a reasonable level) until the evaporating dish attains apparent dryness (about 20 minutes). Using beaker tongs, remove the beaker and evaporating dish from the flame. Holding it with crucible tongs, place the evaporating dish on a wire gauze and gently flame it for about 3 minutes and allow the dish to cool. Weigh the dish to the at least the nearest 0.01 gram. Again flame the evaporating dish gently for several minutes, cool, and weigh. Repeat the process until successive weighing differences are less than 0.01 g. Calculate the mass percent of NaCl in the saturated solution.

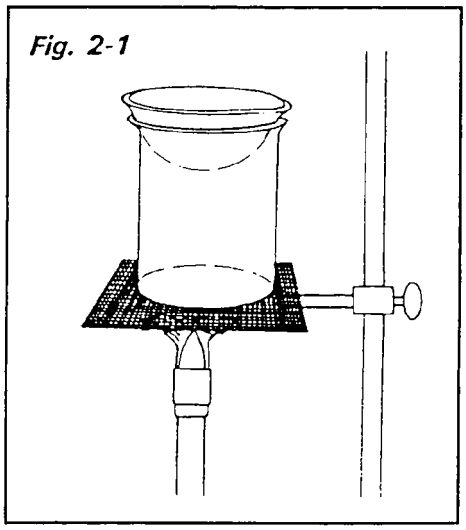


Fig. 2-1

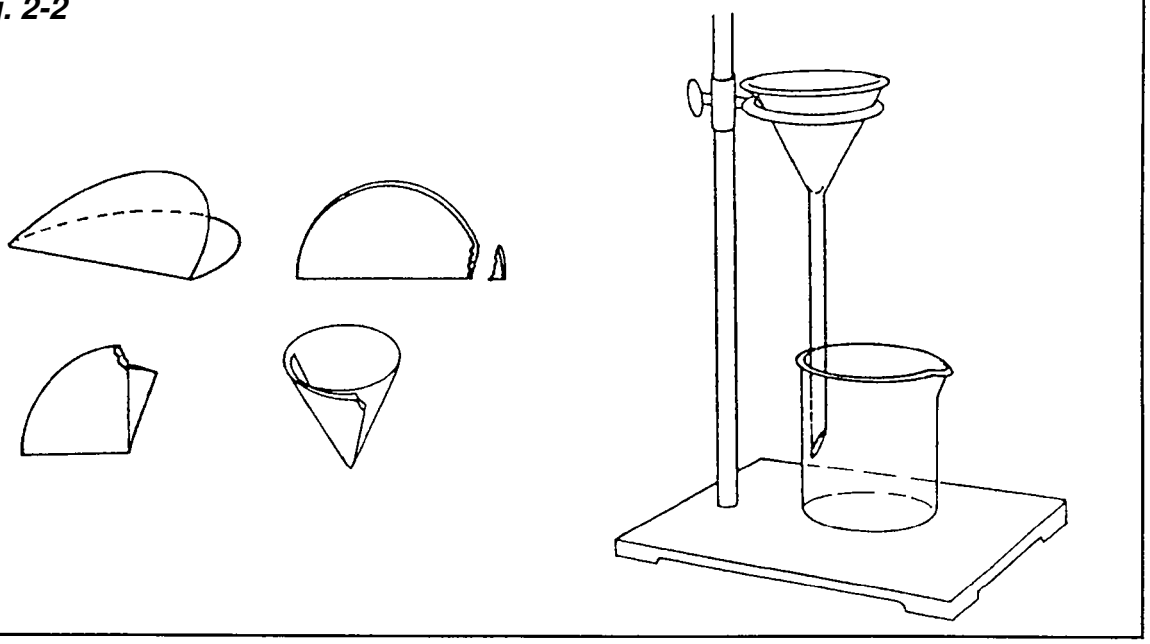
B. Filtration: Collection of calcium carbonate.
The addition of aqueous calcium chloride to aqueous sodium carbonate results in a double replacement reaction or an exchange of the positive and negative partners of two ionic compounds.

$$Na_2CO_{3(aq)} + CaCl_{2(aq)} = 2\,NaCl_{(aq)} + CaCO_{3(s)}$$

As calcium carbonate is not soluble in water, it precipitates out of the solution. The solid calcium carbonate can be separated from the solution by gravity filtration.

Pour about 10 mL of 1.0 M sodium carbonate solution into your graduated cylinder and transfer this solution to a 150 mL beaker. Add 10 mL of 1.0 M calcium chloride solution to the beaker, stir, and report your observations. Save for the filtration.

Fig. 2-2



Select a piece of filter paper that is the appropriate size for your long stem funnel. After being folded and opened into a cone, it should fit slightly below the glass rim of the funnel. For the most common size funnel, this paper will have a 12.5 cm diameter. Fold a piece of filter paper in half. Tear off about a half centimeter piece from one corner then fold it into quarters. Open up one pocket of the filter paper so that it forms the shape of a complete cone. Put it into your funnel and wet it thoroughly with deionized water from your wash bottle so that it adheres uniformly and seals to the inside wall of the funnel. Swirl the reaction mixture and transfer part of it to the filter. Make sure the liquid does not get closer than 0.5 cm to the top edge of the filter paper. Continue adding the mixture until all of it has been added to the funnel. With a wash bottle, squirt some water into the beaker and transfer this wash water to the funnel. Repeat this process two more times. Allow the funnel to drain. When the dripping has virtually stopped, scrape the precipitate onto a watch glass with a stirring rod. Using a medicine dropper, rapidly add about 10 drops of 6 M HCl to the precipitate. Describe your observations.

Whenever a chemical synthesis is attempted (in this case calcium carbonate), it is necessary to verify that the intended product has been isolated. The addition of an acid to a carbonate yields carbon dioxide gas. The observation that a gas evolves provides some evidence but certainly not proof that a carbonate has been synthesized.

Try adding a few drops of 6 M HCl to a small piece of egg shell. Report your observations and conclusions concerning the composition of the egg shell.

C. Recrystallization: Purification of vanillin. The solubility of many solids increases dramatically as the solvent temperature increases. A barely saturated solution of the solid in a hot solvent is prepared and allowed to cool. The solubility decreases as the temperature drops and causes recrystallization to occur. The crystals are collected by vacuum filtration hopefully in a purer condition than before they were dissolved in the solvent. Insoluble impurities are removed by

filtration of the hot saturated solution and soluble impurities stay dissolved in the solvent and do not crystallize upon cooling. These impurities pass through the final cold filtration.

In the following recrystallization of vanillin, recognize that the difficult part of the experiment has already been done for you. For a recrystallization, a solvent must first be found which will dissolve the vanillin when hot but not when cold. Appropriate amounts must also be chosen. Water turns out to be a good solvent for the recrystallization of vanillin and reasonable amounts have been determined by experimentation and reported to you. *Be sure to save the vanillin that you purify today for* ***Experiment 3*** *where you will determine the percent recovery from the recrystallization and the success of the attempted purification.*

Weigh into a 125 or 250 mL Erlenmeyer flask about 2 g of vanillin to the balance limit. Add about 60 mL of deionized water to the flask and stir vigorously. Using a Bunsen burner, heat the solution just to the boiling point and stir until all the solid dissolves. Allow the solution to cool on your desk for several minutes and then put the flask into an ice bath.

Crystallization should occur on its own but occasionally supersaturation occurs and crystallization needs a little assistance. If this happens, try rubbing the inside of the flask beneath the liquid line with a glass rod hard enough to make a grinding sound. If this does not work, seeding with a small crystal of impure vanillin or better yet, a crystal of another student's recrystallized vanillin should initiate the process. After crystallization is complete and the flask has been cooled to about 5°C, isolate the vanillin using vacuum filtration. Obtain a Buchner funnel and filter flask and assemble them as in *Figure 2-3*. Select a piece of filter paper that has been precut to just fit in the bottom of the Buchner funnel.

Connect the filter flask to an aspirator (Note: It is good lab practice to include a trap between the aspirator and the filter flask as water sometimes backs up from the aspirator into the filter flask. However, in this case you are interested in collecting the precipitate only and the filtrate will be discarded. If the filtrate is needed, be sure to use a trap to prevent contamination.) and wet the paper in the filter with deionized water from a wash bottle. Turn the aspirator on full and transfer the contents of the Erlenmeyer to the Buchner funnel. Rinse the Erlenmeyer flask out once or twice with 5 mL of ice-cooled water and use this water to wash the crystals in the funnel. Weigh a piece of filter paper. Empty the crystals onto the weighed filter paper and place them in your desk for drying, being sure they cannot spill. You will weigh the product and check its purity next week.

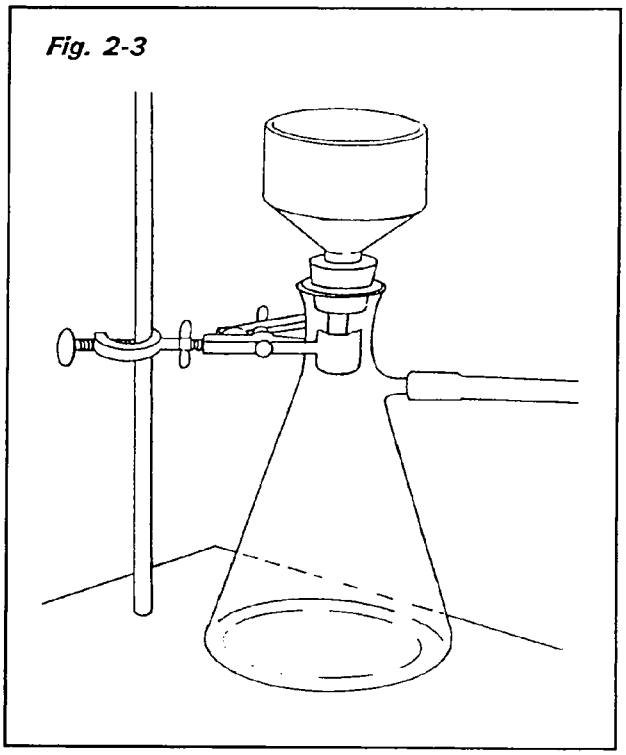


Fig. 2-3

Name_____Date_____Lab Section_____

Prelaboratory Problems - *Experiment 2* - Separation of Mixtures

The solutions to the starred problems are in ***Appendix 4***.

1.* 6.7 mL of a potassium chloride solution was added to a 54.730 g evaporating dish. The combination weighed 61.945 g. After evaporation the dish and contents weighed 55.428 g.

a. What was the mass percent of potassium chloride in the solution? _____

b. If the actual mass percent of potassium in the above solution was 10.00%, what was the percentage error of the above measurement?

_____

c. Why was evaporation used to determine the mass percentage of potassium chloride in the solution rather than filtration or recrystallization? Explain your answer.

d. Compare the mass percent calculated in # 1-c above to the value for a saturated solution in ***Appendix 3***. Was the solution prepared above saturated? Explain your answer.

2. 8.5 mL of a sample of sea water solution was added to a 44.317 g evaporating dish. The combination weighed 52.987 g. After evaporation the dish and contents weighed 44.599 g.

a. What was the mass percent of dissolved solids in the sea water?

_____

b. The actual sodium chloride content in the sea water was 2.69%. If it had been assumed that the dissolved solid(s) consisted only of sodium chloride, what percentage error would have resulted? [Caution: the answer is not 0.56%] The numbers included here are for a typical sea water sample. In addition to NaCl, typical sea water contains about 0.56% by mass of compounds (primarily chlorides) of magnesium, calcium and potassium.

_____

c. Could the dissolved solids have been isolated using either filtration or recrystallization? Explain your answer.

3. Aspirin (acetyl salicylic acid) can be synthesized by reacting acetic anhydride with salicylic acid. Assume the reaction was run and 1.75 grams of a solid product was obtained. Recrystallization of the 1.75 g mixture yielded 1.50 g of aspirin.

a. What was the percentage recovery of aspirin from the recrystallization?

_____

b. Why was recrystallization used for the purification of aspirin rather than filtration or evaporation? Explain your answer.

4. The reaction of aqueous solutions of barium chloride and sodium sulfate results in the formation of a precipitate of barium sulfate. Sodium chloride is also formed but is soluble in water. Why would you use filtration to isolate the barium sulfate rather than evaporation or recrystallization? Explain your answer.

$$BaCl_{2(aq)} + Na_2SO_{4(aq)} = BaSO_{4(s)} + 2\,NaCl_{(aq)}$$

5. Sodium chloride, calcium carbonate and vanillin are three of the chemicals encountered in this experiment. Name some applications of these chemicals around the house.

Name_____Date_____Lab Section_____

Results and Discussion - *Experiment 2* - Separation of Mixtures

A. Evaporation: Determination of the mass percent of sodium chloride in a saturated sodium chloride solution.

1. Mass of evaporating dish _____
2. Mass of dish and sodium chloride solution _____
3. Mass of dish and sodium chloride (1st heating) _____
4. Mass of dish and sodium chloride (2nd heating) _____
5. Mass of dish and sodium chloride (3rd heating if necessary) _____
6. Mass of saturated sodium chloride solution _____
7. Mass of sodium chloride _____
8. Mass percent of sodium chloride in saturated solution _____
9. Calculate the percentage deviation between your value and an accepted literature value of 26.4%. _____
10. Is sea water close to being a saturated sodium chloride solution? Explain your answer [Hint: See ***Prelaboratory Problem*** #2-b in this experiment.]. _____

11. How could you tell that the original salt solution was saturated?

B. Filtration: Collection of calcium carbonate.

1. What did you observe when you mixed solutions of sodium carbonate and calcium chloride? _____

2. What did you observe when you added HCl to the product of your reaction? _____

3. What conclusion did you draw from your observations in #2?

4. What did you observe when you added HCl to a piece of egg shell? _____

5. What conclusion did you come to about the egg shell from your observations in #4?

6. Buildings and statues contain significant percentages of carbonates. Based on your observations in #2 and #4, what effects could acid rain have on these structures?

C. Recrystallization: Purification of vanillin.

1. Mass of crude vanillin (also enter on page 41) _____

2. Mass of paper used for storing vanillin (also enter on page 41) _____

3. Why is the vanillin solution cooled in an ice bath before vacuum filtration?

4. What additional step could be added to this procedure to remove impurities insoluble in the solvent? _____

5. Some of the *Learning Objectives* of this experiment are listed on the first page of this experiment. Did you achieve the *Learning Objectives*? Explain your answer.

Name_____Date_____Lab Section_____

Postlaboratory Problems - *Experiment 2* - Separation of Mixtures

(Note: It might be useful to refer to the solubility chart in ***Appendix 3*** for some of these questions.)

A. Evaporation: Determination of the mass percent of sodium chloride in a saturated sodium chloride solution.

1. What criteria do you think were used for the selection of this system?

   a. Why was water chosen for the solvent?

   b. Why was sodium chloride chosen for the solute?

   c. Suggest reasons why the following were not chosen for the solute:

   calcium carbonate ($CaCO_3$)

   sodium cyanide (NaCN)

   gold chloride ($AuCl_3$)

   hydrogen chloride (HCl)

2. For the evaporation of NaCl, why is the heat-cool-weigh cycle repeated until constant mass is attained?

3. Would evaporation be a very useful separation technique for solutions that contain more than one solute? Explain your answer.

B. Filtration: Collection of calcium carbonate.

Why was gravity filtration used instead of evaporation to isolate the calcium carbonate (consider question *A-3* above)?

C. Recrystallization: Purification of vanillin.

1. What criteria do you think were used to select the solute and solvent for this experiment?

2. a. Suggest any limitations to the use of recrystallization as a purification method for a solid.

   b. Suggest reasons why recrystallization from water would probably not be a suitable procedure for purifying the following substances:

   sodium chloride (NaCl) (Hint: Look up its solubility and also the dependence of the solubility on temperature in your textbook.)

   calcium carbonate ($CaCO_3$)

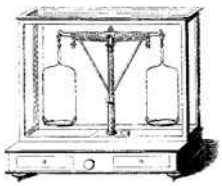
   ethanol (C_2H_6O)

3. Gravity and vacuum filtration separate insoluble solids from a liquid phase. The choice depends on conditions. Suggest criteria you would apply to choose between them.

4. What happens to the salt concentration in a saturated solution if the water is allowed to evaporate (assume constant temperature is maintained)? Explain your answer.

5. Suggest any ways you can think of to improve any part(s) of this experiment.

Experiment 3



MEASUREMENTS AND IDENTIFICATION TECHNIQUES

Learning Objectives

Upon completion of this experiment, students will have experienced:

1. The determination of the percent recovery and purity of recrystallized vanillin (from ***Experiment 2***).
2. The determination of melting points using the capillary technique and the effects of impurities on melting points.
3. The determination of the density of a solid using two methods.

Text Topics

Significant figures, melting points, density (for correlation to some texts, see p. ix).

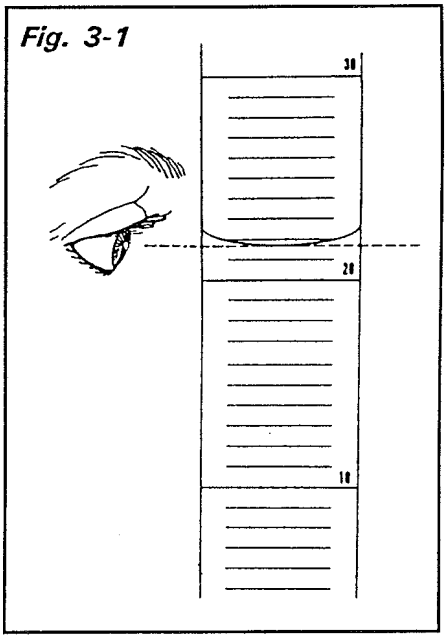
Notes to Students and Instructor

With proper preparation, this experiment and part of the next one can be performed in one laboratory period.

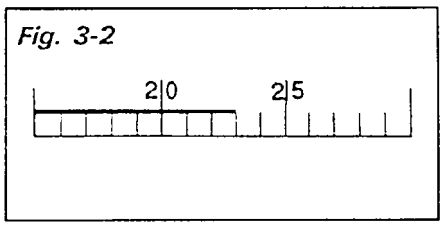
Discussion

The importance of careful and complete descriptive observations cannot be overstated. Often, however, it is desirable to seek quantitative relationships between variables. To accomplish this, we need to perform measurements using a consistent set of units. We know that the volume of a gas will increase as the temperature is increased but can we predict how much it will increase? By measuring the volume of a gas as a function of temperature, it is possible to develop an empirical relationship between volume and temperature. Application of the kinetic theory yields the same equation providing support for the validity of the theory.

For units, scientists have joined most of the rest of the world in adopting the convenient and rational metric system. Prefixes relate different length, mass and volume systems by powers of ten (rather than factors of 3, 12, 16, 36 or 5280). Volume is defined in terms of distance cubed (1 L = 1 dm^3 therefore 1 mL = 1 cm^3) instead of independently (how many gallons in 1 ft^3?). The mass scale has been established so that at 3.98°C, water has a density of exactly 1 g/cm^3 or 1 g/mL (What is the density of water in lbs/ft^3 or lbs/gal?). The metric temperature scale is in Celsius degrees with the freezing and boiling points of water logically defined as 0°C and 100°C respectively.



This experiment will focus on techniques of reading, recording, and utilizing measurements. **One of the most important rules to remember is that you should estimate and record 1 digit beyond the last set of graduations on your measuring instrument** (this rule doesn't apply to many of the new high tech instruments that have digital readout although sometimes fluctuation in the last digit is observed and you then do estimate the most probable value of that digit). The estimated digit does have significance and is therefore counted as a ***significant figure***. In *Figure 3-1*, the reading should be recorded as 21.7 mL and not 22 mL. The bottom of the meniscus (see ***Experiment 1***) is obviously below 22.0 mL and above 21.5 mL. To write down 22 mL would be communicating that the value is between 21 and 23 mL. But it is clear that the reading provides more information than this. The value 21.7 mL communicates to the reader that the value is probably between 21.6 and 21.8 mL. A second rule is that ***when the estimated digit is a zero*** (this should happen about 1 out of every 10 readings), ***the zero must be recorded***. The thermometer below reads 23.0°C. 23°C is an inadequate recording of the temperature as it states that the reading is between 22 and 24°C but observation indicates that it is between 22.9 and 23.1°C



Be especially careful with zeros when a decimal is not showing. If you say you are driving 90 km/hr, do you mean between 89 and 91 or between 80 and 100 (the former should be written 9.0×10^1 km/hr and the latter either 90 km/hr or preferably 9×10^1 km/hr). For more information on measurements, significant figures and rounding off, refer to your textbook.

Intensive Physical Properties. Whether dealing with an unknown chemical extracted from a plant or the product of a chemical synthesis, chemical identification can often pose a significant challenge to the chemist. For previously characterized compounds, a comparison of properties of the unknown with those of knowns will usually make an identification possible. To be useful for identification purposes, a property must not depend on the amount of substance. Extensive properties such as mass and volume do depend on the amount of substance and are not useful for identification purposes. However, intensive properties such as melting points and densities do not depend on the amount of material and are very useful for identification purposes. Melting points of substances are significantly depressed by many additives or impurities. Thus, in addition to being useful for identification purposes, melting points are useful in determining purity.

The density (mass/volume) of a substance is also an intensive property. 1.00 mL of mercury has a mass of 13.6 grams and a density of 13.6 g/mL. 10.0 mL of mercury has a mass of 136 grams and a density of 13.6 g/mL. Although density does require two measurements, mass and volume, both are relatively easy to measure. Comprehensive tables of densities and melting points have been compiled and both are commonly used for identification purposes.

Procedure

A. Percent recovery. Determine the percent recovery of vanillin by determining the mass of the vanillin obtained from the recrystallization in ***Experiment 2***, dividing the amount by the original amount of crude vanillin and then multiplying by 100%. When you are finished with the vanillin, in *Parts A and B,* return your recrystallized vanillin to a stockroom bottle as the vanillin can be used again in ***Experiment 2***.

B. Melting points. The melting point of a pure substance can be measured quickly using the capillary method (described later). The method is accurate to within a few tenths of a degree and requires only a small amount of sample. For identification purposes, the determination of the melting point usually narrows down the number of possible compounds to two or three. When a contaminated compound is melted, we should notice two distinguishing features. First, as with ice water contaminated by salt, the melting point is lowered by additives. Second, the melting process occurs over more than a few tenths of a degree range and is more properly called a melting range than a melting point. **It is very important that the total range be reported; that is the temperature at which the first minute amount of liquid appears to the temperature at which the sample is totally liquid.** The extent of depression and broadening of the melting range serves as a useful measure of the purity of a sample. Very roughly each 1% of impurity (up to about 10%) will depress the high value of the range about 1° below the melting point of the pure compound. Utilizing the melting point depression phenomenon, it is even possible to distinguish between two possible compounds with the same melting points if labeled samples of the two are available (See ***Prelaboratory Problem 5***).

The capillary technique is illustrated in *Figures 3-3* and *3-4*. Select a thermometer that covers the range -10°C to 110°C. Capillary tubes are very convenient sample holders as they are inexpensive and hold very small amounts of sample that can be easily observed during the melting process. Fill the capillary tube by pressing the open end onto the powdered sample until there is about a 0.5-1 cm length of sample in the tube. Now drop the capillary tube, sealed end down, through a 1 meter piece of 6 mm glass tubing that is being held on a hard surface. The impact of the capillary with the hard surface seldom results in breaking and causes the sample to drop to the bottom of the tube. Repeat the dropping procedure until the sample is packed in the bottom of the tube. Attach the capillary tube with a rubber ring (cut off a piece of rubber hose) to a thermometer with the sample even with the bulb of the thermometer. Place the thermometer in a 250 mL beaker half full of water mounted above a Bunsen burner. Support the thermometer using a ***split*** rubber stopper supported by a clamp on a ring stand. Gently heat the water with **continual** stirring and observe the sample. As you approach the melting range, slow the heating rate to around 2°C/min. Heating the sample too rapidly near its melting range will result in large errors. At the first indication of sample melting, record the temperature to the nearest 0.1°C. Continue to slowly heat until the sample has totally liquified and record the high end of the melting range.

Using the technique described above, determine the melting ranges of the three samples below. [Hint: The melting ranges should occur between 60°C and 85°C. As the melting range is approached, the heating rate should be kept as slow as possible.] For the mixed sample, add approximately equal amounts of each compound to a mortar and grind them together with a pestle before inserting into a capillary tube. Don't forget to recycle your vanillin.

a. Impure vanillin from ***Experiment 2.***
b. Recrystallized vanillin from ***Experiment 2.***
c. 50% recrystallized vanillin + 50% phenyl carbonate

Fig. 3-3

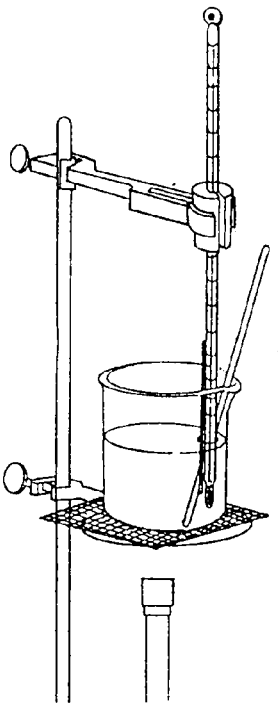
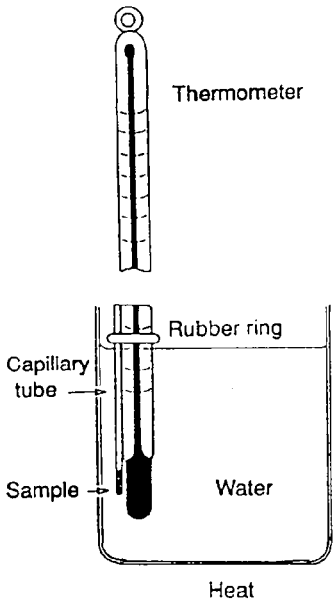


Fig. 3-4



C. Density. The density, or mass/volume, of a metal cylinder will be calculated twice using two different methods to determine its volume. Then you will use the density to attempt to identify the metal. First use your balance to determine the mass of the metal cylinder to the limits of the balance (probably either 0.01 g or 0.001 g.).

1. Volume from linear measurements: For regularly shaped objects such as a cylinder, it is usually possible to measure the dimensions of the object and use a formula to calculate the volume of the object. The volume of a cylinder is calculated by multiplying π times the square of its radius times its length ($V = \pi r^2 L$). Using a metric ruler or preferably vernier calipers, measure the diameter and length of the cylinder and calculate its volume and density.

2. Volume from water displacement: A more general method for volume determination that will work with regular and irregular shaped objects is the method of water displacement. The water level in a graduated cylinder is recorded, an object submerged in the water and the new water level read. The difference in water levels is the volume of the object. Obtain an appropriately sized graduated cylinder for your metal cylinder and fill it about half full with water. Read the water level, tilt the graduated cylinder and slide the metal cylinder gently into the water being sure that it is completely submerged. Read the resulting water level and calculate the volume and density of the object.

3. Assume the metal cylinder is one of the following: aluminum, copper, iron or lead. Look up the density of each metal and determine the composition of your cylinder.

Name_____Date_____Lab Section_____

Prelaboratory Problems - *Experiment 3* - Measurement and Identification

The solutions to the starred problems are in ***Appendix 4***.

1. Math review problems

   a.* How many significant figures are in each of the following numbers?

   405.0 _____ 0.0789 _____ 2.040 _____ 360 _____ 3.00×10^{10} _____

   b. Express each of the following with three significant figures.

   0.002537 _____ 12345000 _____ 0.07966 _____ 620.2 _____

   c. Perform the following operations: 4.5×10^{3} cm + 1.6×10^{2} cm = _____

   $(1.5 \times 10^{-3} \text{ mole})\dfrac{(6.022 \times 10^{23} \text{ molecules})}{1 \text{ mole}}$ = _____ $\dfrac{1.80 \times 10^{-6} \text{ g}}{2.00 \times 10^{-3} \text{ mL}}$ = _____

2. List at least four distinct advantages of the metric system over "our" measurement system.

3. a.* Salt is often spread on icy roads in the winter. What is its function and how does it work?

   b. Due to the use of salt on roads, two towns in Massachusetts were having problems with high salt content in ground water supplies. One town chose not to use salt one winter and the automobile accident rate actually went down. Suggest a reason for this unexpected result and comment on the validity of long range conclusions from this study.

   c. When preparing home-made ice cream, salt is added to the ice-water mixture. What is its function and why does it work?

   d. Antifreeze (commonly ethylene glycol or propylene glycol) is usually mixed with water in the radiator of a car. What is its function and how does it work?

4.* A 2.5 g sample of naphthalene (melting range 73.4-77.1°C) is recrystallized and 1.8 grams of purified naphthalene (melting range 80.5-80.6°C) are recovered.

a. What is the percent recovery? _____

b. Comment on the purity of the original naphthalene sample.

c. Comment on the success of the recrystallization.

5. Cinnamic acid and urea melt at 133°C. The melting point of an unknown is found to be 133°C. What experiments using melting range determinations could be done to determine if the unknown is cinnamic acid or urea?

6.* A 15.00 g metal sphere was found to have a diameter of 1.85 cm. The volume of a sphere is $V = (4/3)\pi r^3$. Calculate the density of the sphere and assuming that the sphere is made out of one of the elements, aluminum, chromium, iron, lead, titanium or zinc, determine its composition.

7. A 55.81 gram irregular object made out of one of the metals in *#6* raises the water level in a graduated cylinder from the 17.6 mL level to the 24.7 mL level. What is the density and identity of the metal?