STUDENT EDITION

CHEMISTRY SMALL-SCALE LABORATORY MANUAL



A GLENCOE PROGRAM



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To the Student

Chemistry is the science of matter, its properties, and changes. In your classroom work in chemistry, you will learn a great deal of the information that has been gathered by scientists about matter. But chemistry is not just information. It is also a process for finding out more about matter and its changes. Laboratory activities are the primary means that chemists use to learn more about matter. The activities in the *Chemistry Small-Scale Laboratory Manual* require that you form and test hypotheses, measure and record data and observations, analyze those data, and draw conclusions based on those data and your knowledge of chemistry. These processes are the same as those used by professional chemists and all other scientists.

Chemistry Small-Scale Laboratory Manual activities use the latest development in laboratory techniques—small-scale chemistry. In small-scale chemistry, you often use plastic pipettes and microplates instead of large glass beakers, flasks, and test tubes. You also use small amount of chemicals in reactions. Still, when working with small-scale chemistry, you should use the same care in obtaining data and making observations that you would use in large-scale laboratory activities. Likewise, you must observe the same safety precautions as for any chemistry experiment.

Organization of Activities

- **Introduction** Following the title and number of each activity, an introduction provides a background discussion about the problem you will study in the activity.
- **Problem** The problem to be studied in this activity is clearly stated.
- **Objectives** The objectives are statements of what you should accomplish by doing the investigation. Recheck this list when you have finished the activity.
- Materials The materials list shows the apparatus you need to have on hand for the activity.
- **Safety Precautions** Safety symbols and statements warn you of potential hazards in the laboratory. Before beginning any activity, refer to page vii to see what these symbols mean.
- **Pre-Lab** The questions in this section check your knowledge of important concepts needed to complete the activity successfully.
- **Procedure** The numbered steps of the procedure tell you how to carry out the activity and sometimes offer hints to help you be successful in the laboratory. Some activities have **CAUTION** statements in the procedure to alert you to hazardous substances or techniques.
- **Hypothesis** This section provides an opportunity for you to write down a hypothesis for this activity.
- Data and Observations This section presents a suggested table or form for
 collecting your laboratory data. Always record data and observations in an organized way as you do the activity.
- Analyze and Conclude The Analyze and Conclude section shows you how to perform the calculations necessary for you to analyze your data and reach conclusions. It provides questions to aid you in interpreting data and observations in order to reach an experimental result. You are also asked to form a scientific conclusion based on what you actually observed, not what "should have happened." An opportunity to analyze possible errors in the activity is also given.
- **Real-World Chemistry** The questions in this section ask you to apply what you have learned in the activity to other real-life situations. You may be asked to make additional conclusions or research a question related to the activity.

Small-Scale Laboratory Techniques

Small-scale chemistry uses smaller amounts of chemicals than do other chemistry methods. The hazards of glass have been minimized by the use of plastic labware. If a chemical reaction must be heated, hot water will provide the needed heat. Open flames or burners are seldom used in microchemistry techniques. By using small-scale chemistry, you will be able to do more experiments and have a safer environment in which to work.

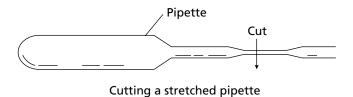
Small-scale chemistry uses two basic tools.

The Microplate

The first is a sturdy plastic tray called a microplate. The tray has shallow wells arranged in rows (running across) and columns (running up and down). These wells are used instead of test tubes, flasks, and beakers. Some microplates have 96 wells; other microplates have 24 larger wells.

The Plastic Pipette

Small-scale chemistry uses a pipette made of a form of plastic that is soft and very flexible. The most useful property of the pipette is the fact that the stem can be stretched without heating into a thin tube. If the stem is stretched and then cut with scissors, the small tip will deliver a tiny drop of reagent. You may also use a pipette called a microtip pipette, which has been pre-stretched at the factory. It is not necessary to stretch a microtip pipette.



The pipette can be used over and over again simply by rinsing the stem and bulb between reagents. The plastic inside the pipette is non-wetting and does not hold water or solutions the way glass does.

The Microplate Template and Microplate Data Form

Your teacher can provide you with Microplate Templates and Microplate Data Forms whenever you carry out an activity that requires them.

To help you with your observations, place the Microplate Template beneath your 24-well or 96-well microplate. The template is marked with the correct number of wells, and each row and column is labeled to help guide you with your placement of chemicals from the micropipettes. The white paper background provided by the template allows you to observe color changes and precipitate formations with ease.

Use Microplate Data Forms to write down the chemicals used and to record your observations of the chemical reactions that occur in each well.

Waste Disposal

Discard all substances according to your teacher's instructions. All plastic small-scale chemistry equipment can be washed with distilled water for reuse.

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Safety in the Laboratory

The chemistry laboratory is a place to experiment and learn. You must assume responsibility for your own personal safety and that of people working near you. Accidents are usually caused by carelessness, but you can help prevent them by closely following the instructions printed in this manual and those given to you by your teacher. The following are some safety rules to help guide you in protecting yourself and others from injury in a laboratory.

- **1.** The chemistry laboratory is a place for serious work. Do not perform activities without your teacher's permission. **Never** work alone in the laboratory. Work only when your teacher is present.
- **2.** Study your lab activity **before** you come to the lab. If you are in doubt about any procedures, ask your teacher for help.
- **3.** Safety goggles and a laboratory apron must be worn whenever you work in the lab. Gloves should be worn whenever you use chemicals that cause irritations or can be absorbed through the skin.
- **4.** Contact lenses should not be worn in the lab, even if goggles are worn. Lenses can absorb vapors and are difficult to remove in an emergency.
- **5.** Long hair should be tied back to reduce the possibility of it catching fire.
- **6.** Avoid wearing dangling jewelry or loose, draping clothing. The loose clothing may catch fire and either the clothing or jewelry could catch on chemical apparatus.
- **7.** Wear shoes that cover the feet at all times. Bare feet or sandals are not permitted in the lab.
- **8.** Know the location of the fire extinguisher, safety shower, eyewash, fire blanket, and first-aid kit. Know how to use the safety equipment provided for you.
- **9.** Report any accident, injury, incorrect procedure, or damaged equipment immediately to your teacher.
- **10.** Handle chemicals carefully. *Check the labels of all bottles before removing the contents.* Read the labels three times: before you pick up the container, when the container is in your hand, and when you put the bottle back.
- **11.** Do **not** return unused chemicals to reagent bottles.
- **12.** Do **not** take reagent bottles to your work area unless specifically instructed to do so. Use test

- tubes, paper, or beakers to obtain your chemicals. Take only small amounts. It is easier to get more than to dispose of excess.
- **13.** Do **not** insert droppers into reagent bottles. Pour a small amount of the chemical into a beaker.
- **14. Never** taste any chemical substance. **Never** draw any chemicals into a pipette with your mouth. Eating, drinking, chewing gum, and smoking are prohibited in the laboratory.
- **15.** If chemicals come into contact with your eyes or skin, flush the area immediately with large quantities of water. Immediately inform your teacher of the nature of the spill.
- **16.** Keep combustible materials away from open flames. (Alcohol and acetone are combustible.)
- **17.** Handle toxic and combustible gases only under the direction of your teacher. Use the fume hood when such materials are present.
- **18.** When heating a substance in a test tube, be careful not to point the mouth of the tube at another person or yourself. Never look down the mouth of a test tube.
- **19.** Use caution and the proper equipment when handling hot apparatus or glassware. Hot glass looks the same as cool glass.
- **20.** Dispose of broken glass, unused chemicals, and products of reactions only as directed by your teacher.
- **21.** Know the correct procedure for preparing acid solutions. *Always add the acid slowly to the water*.
- **22.** Keep the balance area clean. Never weigh chemicals directly on the pan of the balance.
- **23.** Do **not** heat graduated cylinders, burettes, or pipettes with a laboratory burner.
- **24.** After completing an activity, clean and put away your equipment. Clean your work area. Make sure the gas and water are turned off. Wash your hands

Chemistry uses safety symbols to alert you to possible laboratory dangers. These symbols are provided in the textbook and are explained below. Be sure you understand each symbol before you begin an activity that displays a symbol.

SAFETY SYMBOLS EXAMPLES PRECAUTION HAZARD REMEDY Special disposal procertain chemicals, Do not dispose of Dispose of wastes as **DISPOSAL** cedures need to be living organisms these materials in the directed by your followed. sink or trash can. teacher. Organisms or other Avoid skin contact Notify your teacher if bacteria, fungi, blood, **BIOLOGICAL** biological materials unpreserved tissues, with these materials. you suspect contact that might be harmful plant materials Wear mask or gloves. with material. Wash hands thoroughly. to humans Objects that can burn boiling liquids, hot Use proper protection Go to your teacher for **EXTREME** skin by being too cold plates, dry ice, liquid when handling. first aid. **TEMPERATURE** or too hot nitrogen Use of tools or glassrazor blades, pins, Practice common-Go to your teacher for **SHARP** scalpels, pointed ware that can easily sense behavior and first aid. **OBJECT** tools, dissecting puncture or slice skin follow guidelines for use of the tool. probes, broken glass Possible danger to ammonia, acetone, Make sure there is Leave foul area and **FUME** respiratory tract from nail polish remover, good ventilation. notify your teacher Never smell fumes fumes heated sulfur, moth immediately. balls directly. Wear a mask. Possible danger from improper grounding. Double-check setup Do not attempt to fix **ELECTRICAL** electrical shock or liquid spills, short with teacher. Check electrical problems. burn circuits, exposed condition of wires and Notify your teacher apparatus. immediately. wires Substances that can pollen, moth balls, Wear dust mask and Go to your teacher for **IRRITANT** gloves. Practice extra irritate the skin or steel wool, fiberglass, first aid. mucous membranes potassium permancare when handling of the respiratory tract these materials. ganate bleaches such as Chemicals that can Wear goggles, gloves, Immediately flush the CHEMICAL react with and destroy hydrogen peroxide: affected area with and an apron. acids such as sulfuric tissue and other matewater and notify your rials acid, hydrochloric teacher. acid: bases such as ammonia, sodium hydroxide Substance may be mercury, many metal Follow your teacher's Always wash hands TOXIC poisonous if touched. compounds, iodine, instructions. thoroughly after use. inhaled, or swallowed Go to your teacher for poinsettia plant parts first aid. Open flame may ignite alcohol, kerosene. Tie back hair. Avoid Notify your teacher flammable chemicals, potassium permanwearing loose clothing. immediately. Use fire loose clothing, or hair ganate, hair, clothing Avoid open flames safety equipment if when using flammable applicable. chemicals. Be aware of locations of fire safety equipment.

Radioactivity

when radioactive

materials are used.

This symbol appears



Clothing

clothing.

Protection

This symbol appears

when substances

could stain or burn

Eye Safety

activities.

Proper eye protec-

tion should be worn

at all times by any-

one performing or

observing science

Handwashing

goggles.

After the lab, wash

hands with soap and

water before removing

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Small-Scale Laboratory Techniques

early all experiments in chemistry involve making measurements of some sort. Measurements allow chemists to collect quantitative information about the phenomena they study, such as how much of a substance is present, what its temperature is, or how quickly it was produced. The equipment and techniques used to make scientific measurements vary with the type of information that is being collected.

Problem

What techniques are used to make a dilute solution of candy in water?

Objectives

- Measure the mass of a piece of candy.
- Measure the volume of a small amount of water.
- **Dissolve** the candy in the water.
- Use a pipette and a microplate to make serial dilutions of the candywater solution.

Materials

100-mL beaker 25-mL graduated cylinder 24-well microplate candy balance mortar and pestle spatula thin-stem pipette sheet of white paper

Safety Precautions



• Always wear safety goggles and a lab apron.

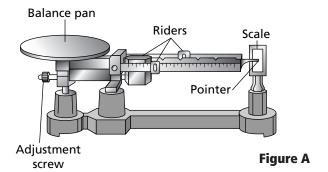
Pre-Lab

- **1.** What is the SI base unit for mass?
- **2.** What quantity is measured in milliliters?
- **3.** How many milliliters are in 1 liter? In 20 cubic centimeters?

Procedure

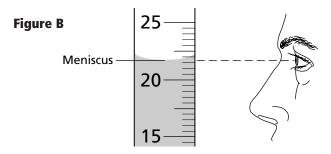
Part A: Measuring Mass

- **1.** Slide all the riders on the balance as far to the left as they will go, as shown in **Figure A.** Check that the pointer swings freely along the scale.
- 2. With nothing on the balance pan, the pointer should swing an equal distance above and below the zero mark on the scale. If it does not, turn the adjustment screw until the swings above and below zero are equal.



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- **3.** Gently set the beaker on the balance pan. Notice that the pointer moves to the top of the scale.
- **4.** Beginning with the largest rider on the top beam, move the riders to the right until the pointer again swings an equal distance above and below the zero mark. If the beams are notched, make sure each rider rests in a notch.
- **5.** To find the mass of the beaker, add the masses indicated on the beam riders. Record the mass of the beaker to the nearest 0.1 g in **Data Table 1.**
- **6.** Place one piece of candy in the beaker. Reposition the riders until the pointer again swings an equal distance above and below the zero mark. Record the mass of the beaker plus candy to the nearest 0.1 g in **Data Table 1.**



Part B: Measuring Volume

- 1. Pour about 20 mL of water into the graduated cylinder. As **Figure B** shows, the water in the cylinder has a curved surface, called a *meniscus*. To take a volume reading, view the bottom of the meniscus at eye level. Unless this position lines up exactly with a marking on the cylinder, you will need to estimate the distance between two markings.
- 2. The volume of water in the cylinder is measured by the closest marking on the side of the cylinder that lines up with the bottom of the meniscus. Record the volume to the nearest 0.1 mL in Data Table 1.

Part C: Making a Solution

1. Grind the candy into small pieces with the mortar and pestle, as shown in **Figure C.** (Candy should NOT be reduced to a fine powder.) Use the spatula to scrape the ground candy back into the beaker.

Figure C

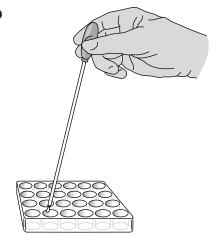


- **2.** If any candy remains in the mortar, pour some water from the graduated cylinder into the mortar. Grind the remaining candy in the mortar until it dissolves in the water. Pour this solution into the beaker.
- **3.** Add the rest of the water to the beaker. Swirl the beaker until all of the pieces of candy are dissolved.

Part D: Making Serial Dilutions

- Fill the graduated cylinder with water. Use the pipette to place 10 drops of water in the top left well of the microplate, as shown in Figure D. Notice that this well is labeled A1. CAUTION: Never place the pipette in your mouth.
- **2.** Place 10 drops of the candy-water solution in well B1.
- **3.** Transfer 1 drop of the candy-water solution from well B1 to well B2. Add 9 drops of water from the graduated cylinder to well B2. Well B2 now contains a diluted candy-water solution.

Figure D





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- **4.** Place a sheet of white paper beneath the microplate. Compare the color of the contents of wells A1, B1, and B2.
- **5.** Transfer 1 drop of the diluted candy-water solution to the next well in row B. Add 9 drops of water to that well. Compare the color of the new solution to that of the others.
- **6.** Repeat step 5 until the most dilute solution appears completely colorless.

Cleanup and Disposal

1. Make sure your balance is left in the same condition as you found it and all riders are set to zero.

- 2. Return all lab equipment to its proper place.
- **3.** Dispose of the candy-water solutions in the sink.
- **4.** Wash your hands thoroughly with soap or detergent before you leave the lab.

Data and Observations

Data Table	e 1
Mass of beaker (g)	
Mass of beaker + candy (g)	
Mass of candy (g)	
Volume of water (mL)	

Analyze and Conclude

- **1. Measuring and Using Numbers** To find the mass of candy, subtract the mass of the beaker from the mass of the beaker + candy. Record the result in **Data Table 1.**
- **2. Measuring and Using Numbers** Calculate the concentration of candy in the candy-water solution in well B1. (Hint: The answer should have units of g/mL.)
- **3. Thinking Critically** Calculate the concentration of candy in the diluted candy-water solution in well B2. (Hint: Remember that you added 9 drops of water to 1 drop of the undiluted candy-water solution.)
- **4. Measuring and Using Numbers** What was the concentration of candy in the most dilute solution you made (the one that appeared completely colorless)?

Real-World Chemistry

Ammonia solution is a household cleaner with many uses. To clean windows, you can prepare a diluted solution by mixing 1 tablespoon of ammonia solution with 1 quart of water. If the undiluted solution contains 10 percent ammonia, what percent of the diluted solution is ammonia? (Hint: 1 tablespoon = 15 mL, and 1 quart = 0.95 L)

Class

LAB



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Comparing the Density of Metals

Different materials can be distinguished from one another because they have different properties. One property that is often used to identify unknown materials is density. Density is defined as the ratio of a material's mass to its volume. By measuring the mass and volume of a sample of material, you can obtain an important clue about the identity of the material.

Problem

Can you identify unknown metals by calculating their densities?

Objectives

- Measure the mass and volume of four metal samples.
- Calculate the density of each sample from these measurements.
- Compare the calculated densities with known densities of specific metals.
- **Identify** each metal sample.

Materials

metal samples
balance
50-mL graduated
cylinder
water
paper towel
CRC Handbook of
Chemistry and
Physics (optional)

Safety Precautions









Always wear safety goggles and a lab apron.

Pre-Lab

- **1.** What is the formula to calculate density? What are the units for density?
- **2.** Explain the difference between base units and derived units.
- **3.** Is density measured in base units or derived units?
- **4.** A sample of metal X has a mass of 85.6 g and a volume of 12.1 mL. What is the density of metal X?
- **5.** A metal bar has a density of 19.3 g/mL and a mass of 50.0 kg. What is the volume of the bar?

Procedure

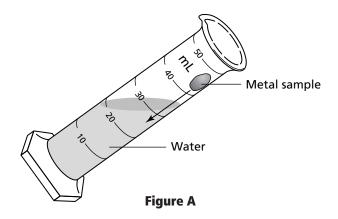
- **1.** Select a metal sample from the materials table. Record the letter of the sample in **Data Table 1.**
- **2.** Use the balance to measure the mass of the sample to the nearest 0.01 g. Record the mass in **Data Table 1.**
- **3.** Fill the graduated cylinder half full of water. If there are air bubbles in the water, tap the outside of the cylinder with your finger to remove them. Record the volume of water to the nearest 0.1 mL.

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- **4.** Tilt the cylinder and carefully insert the metal sample. Let the sample slide down the cylinder without splashing any water, as shown in **Figure A.** Make sure that the sample is completely under water and that there are no air bubbles. Then record the volume of water plus metal sample.
- **5.** Repeat steps 1–4 for the other three metal samples.

Cleanup and Disposal

- **1.** Dry the metal samples with a paper towel and return them to the materials table.
- **2.** Make sure your balance is left in the same condition as you found it.
- **3.** Return all lab equipment to its proper place, as directed by your teacher.



Data and Observations

	Data Table 1							
Sample	Mass (g)	Volume of water (mL)	Volume of water + sample (mL)	Volume of sample (mL)	Density (g/mL)			

- **1.** To find the volume of each metal sample, subtract the volume of water from the volume of water + sample. Record the results in **Data Table 1.**
- 2. To calculate the density of each metal sample and record the results in **Data Table 1**.

Analyze and Conclude

1.	Acquiring and Analyzing Information Look up the densities of the following metals:
	aluminum, copper, iron, lead, tin, tungsten, and zinc.

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2.	Drawing Conclusions Assume that the samples you tested may be any of the metals in question 1. Decide which metal each sample is likely to represent.
3.	Applying Concepts If there were air bubbles in the water after you added a sample but not before, would that affect the density value you calculated for that sample? Explain.
. .	Thinking Critically Suppose you calculated the density of a metal sample to be 7 g/mL. Describe two ways you could determine whether the sample is made of tin or zinc.
j .	Error Analysis Find out from your teacher whether you correctly identified the samples. Compare the density you calculated for each sample with the accepted density of the metal it is made of. Calculate the percentage error if any. Explain possible sources of error in the lab.

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Real-World Chemistry

- 1. Body composition refers to the percentage of a person's body mass that is composed of lean tissue or fatty tissue. Lean tissue, which includes muscle and bone, is more dense than water. Fatty tissue is less dense than water. Would you expect a person who has 30 percent body fat to float higher or lower in the water than a person of the same mass who has 10 percent body fat? Explain.
- **2.** Modern jet airplanes are built primarily out of aluminum and another metal, titanium, which has a density of 4.5 g/mL. Why do you think these metals are preferred over other metals, such as iron and lead?
- **3.** Many metallic materials are alloys, or mixtures of two or more metals. For example, brass is an alloy of copper and zinc. How would you expect the density of brass to compare to the density of pure copper? Explain.



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Separation of Aspirin

which is commonly called aspirin. An aspirin tablet contains more than aspirin, however. Manufacturers mix aspirin with starch, which keeps the tablets from falling apart and makes them large enough for easy handling. Furthermore, aspirin can break down into salicylic acid and acetic acid over time. Therefore, an aspirin tablet is a mixture of at least four substances: aspirin, starch, salicylic acid, and acetic acid.

Problem

How can you separate the substances in an aspirin tablet?

Objectives

- **Separate** an aspirin tablet into two phases.
- **Test** each phase for the presence of starch.
- **Test** one of the phases for the presence of salicylic acid.
- Compare the amount of salicylic acid in new and old aspirin tablets.

Materials

aspirin tablets
(1 old, 1 new)
isopropyl alcohol
(2-propanol)
iron(III) nitrate
solution
iodine solution

mortar and pestle 24-well microplate thin-stem pipette spatula toothpicks (2) sheet of white paper

Safety Precautions



- Always wear safety goggles, gloves, and a lab apron.
- Use care when handling all solutions.

Pre-Lab

- **1.** What is the difference between a heterogeneous mixture and a homogeneous mixture?
- **2.** Classify the following mixtures as heterogeneous or homogeneous: salt and water, sand and water, nitrogen and oxygen in air.
- **3.** Which technique—distillation, crystallization, or filtration—is most useful for separating a heterogeneous mixture composed of a solid and a liquid?
- **4.** What technique is used to separate the substances in a solution based on differences in their boiling points?
- **5.** Read the entire laboratory activity. Form a hypothesis about whether new or old aspirin tablets will contain more salicylic acid. Explain why. Record your hypothesis on page 10.

Procedure

- **1.** Place a sheet of white paper beneath the 24-well microplate.
- **2.** Grind an aspirin tablet from group A into small pieces with the mortar and pestle. Use the spatula to scrape the ground tablet into well A1 of the microplate.
- **3.** Use the pipette to add 40 drops of isopropyl alcohol to well A1. Stir the mixture in well A1 with a toothpick.
- **4.** Allow the solid material in well A1 to settle to the bottom of the well.

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- 5. Use the pipette to remove the liquid from well A1. CAUTION: Never place the pipette in your mouth. Be careful not to draw up any of the solid material. The liquid consists of isopropyl alcohol and any substances in the aspirin tablet that can dissolve in isopropyl alcohol. Place the liquid in well A2.
- **6.** Repeat steps 3–5.
- **7.** Transfer 10 drops of the liquid in well A2 to well A3.
- **8.** Clean and dry the mortar and pestle.
- **9.** Repeat steps 2–7 using an aspirin tablet from group B and wells B1–B3 of the microplate.
- **10.** Add 1 drop of iodine solution to wells A1, A2, B1, and B2. Stir with a toothpick. Record what happens to the contents of each well in **Data Table 1.**
- **11.** Add 1 drop of iron(III) nitrate solution to wells A3 and B3. Stir with a toothpick. Compare the color of the contents of these wells. Record your observations in **Data Table 1.**

Hypothesis		

Cleanup and Disposal

- **1.** Dispose of all solutions as directed by your teacher.
- **2.** Return all lab equipment to its proper place.
- **3.** Wash your hands thoroughly with soap or detergent before you leave the lab.

Data and Observations

	Data Table 1			
Well	Observations			
A1				
A2				
A3				
B1				
B2				
В3				

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Analyze and Conclude

Observing and Inferring Iodine solution turns blue or black when added to starch. Using this information, can you determine whether starch dissolves in isopropyl alcohol? Explain. Observing and Inferring Iron(III) nitrate solution turns violet when added to salicylic acid. Using this information, can you determine whether salicylic acid dissolves in isopropyl alcohol? Explain. Drawing a Conclusion The more salicylic acid a solution contains, the darker the violet color that results when iron(III) nitrate solution is added. Using this information, compare the amount of salicylic acid in the aspirin tablets from group A and group B. Drawing a Conclusion Which tablet (A or B) was the old sample and which was the new sample? Error Analysis Find out from your teacher which group (A or B) contained old aspirin
Observing and Inferring Iron(III) nitrate solution turns violet when added to salicylic acid. Using this information, can you determine whether salicylic acid dissolves in isopropyl alcohol? Explain. Drawing a Conclusion The more salicylic acid a solution contains, the darker the violet color that results when iron(III) nitrate solution is added. Using this information, compare the amount of salicylic acid in the aspirin tablets from group A and group B. Drawing a Conclusion Which tablet (A or B) was the old sample and which was the new sample?
Drawing a Conclusion The more salicylic acid a solution contains, the darker the violet color that results when iron(III) nitrate solution is added. Using this information, compare the amount of salicylic acid in the aspirin tablets from group A and group B. Drawing a Conclusion Which tablet (A or B) was the old sample and which was the new sample?
color that results when iron(III) nitrate solution is added. Using this information, compare the amount of salicylic acid in the aspirin tablets from group A and group B. Drawing a Conclusion Which tablet (A or B) was the old sample and which was the new sample?
new sample?
new sample?
Error Analysis Find out from your teacher which group (A or B) contained old aspirin
and which contained new aspirin. Then compare the results of this experiment with the predictions of your hypothesis. Explain possible reasons for any disagreement.

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Real-World Chemistry

- 1. Panning is a method for separating gold from the other materials with which it is often mixed. A person using this method fills a circular pan with gold-containing sand or gravel and swirls the pan under a gentle stream of water. Explain why the gold separates from the other materials under these conditions. (Hint: The density of gold is 19.3 g/mL, and the density of sand and gravel ranges from about 2.5 to 5.0 g/mL.)
- 2. Sodium chloride (table salt) is an essential nutrient for humans and other animals. It is also the major substance dissolved in seawater. Describe a simple method for separating sodium chloride from seawater.
- **3.** Many communities have recycling programs for both aluminum cans and for cans made of iron. Some programs ask citizens to keep the two kinds of cans separate, while other programs do not. How might recyclers easily separate aluminum cans from cans made of iron, even if all of the cans are ground up and the pieces are thoroughly mixed together?



CHEMISTRY SMALL-SCALE



Periodicity and the Properties of Elements

he periodic table of elements organizes the elements according to their atomic structures. The table is arranged in horizontal rows called periods and vertical columns called groups or families. Elements in the same group have similar chemical and physical properties. Thus, it is possible to predict many of the properties of an element by examining its position in the table. One such property is solubility. Solubility refers to the amount of a substance that can dissolve in a given amount of another substance. In this activity, you will demonstrate patterns of solubility.

Problem

How can you demonstrate patterns of solubility for compounds containing alkaline earth metals?

Objectives

- Prepare serial dilutions of solutions containing ions of alkaline earth metals.
- **Observe** precipitates that form when other chemicals are added to these solutions.
- Recognize patterns of solubility for compounds containing alkaline earth metals.

Materials

96-well sheets of black microplates (3) construction solutions of: paper (15) $Mg(NO_3)_2$ 96-well $Ca(NO_3)_2$ templates (3) $Sr(NO_3)_2$ thin-stem $Ba(NO_3)_2$ pipettes (7) toothpicks (45) Na₂SO₄ distilled water Na₂C₂O₄ Na₂CO₃

Safety Precautions









- Always wear safety goggles, gloves, and a lab apron.
- Use extra care when handling all solutions.
- Notify your teacher of any spills.
- Dispose of all solutions as directed by your teacher.

Pre-Lab

- **1.** Why do elements in the same group on the periodic table have similar properties?
- **2.** Why don't elements in the same group have identical properties?
- **3.** What is the relationship between reactivity and atomic number for metals in the same group?
- **4.** Name the alkaline earth metals.

5. Read the entire laboratory activity. Form a hypothesis about the solubility of the compounds containing alkaline metals that you will test in this experiment. Record your hypothesis on page 14.

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

Procedure

- **1.** Place a microplate on the construction paper with the lettered rows on the left.
- 2. Using a different pipette for each solution, place 10 drops of Mg(NO₃)₂ in well A1, 10 drops of Ca(NO₃)₂ in well B1, 10 drops of Sr(NO₃)₂ in well C1, and 10 drops of Ba(NO₃)₂ in well D1. CAUTION: Never place the pipette in your mouth.
- **3.** Label all three templates to show the solutions that are in wells A1–D1. Title Template 1 "Precipitate Formed by Adding Na₂SO₄." Title Template 2 "Precipitate Formed by Adding Na₂C₂O₄." Title Template 3 "Precipitate Formed by Adding Na₂CO₃."
- **4.** Add 5 drops of distilled water to each of wells A2–A12, B2–B12, C2–C12, and D2–D12.
- **5.** Transfer 5 drops of the solution in well A1 to well A2. Mix thoroughly with a toothpick.
- **6.** Continue transferring 5 drops from one well to the next through well A12, as diagrammed in **Figure A.**
- **7.** Remove and discard 5 drops of the solution in well A12.
- **8.** Using a different pipette for each row, repeat steps 5–7 for rows B, C, and D.
- **9.** Add 5 drops of Na₂SO₄ to each well that contains a solution.
- **10.** Examine each well for the presence of a precipitate (solid material at the bottom of the well). Indicate which wells contain a precipitate on Template 1.
- **11.** Using a clean microplate, repeat steps 2–8. You may use the same pipettes you used before to transfer the solutions.

- **12.** Add 5 drops of Na₂C₂O₄ to each well that contains a solution.
- **13.** Examine each well for the presence of a precipitate. Indicate which wells contain a precipitate on Template 2.
- **14.** Using a clean microplate, repeat steps 2–8. You may use the same pipettes you used before to transfer the solutions.
- **15.** Add 5 drops of Na₂CO₃ to each well that contains a solution.
- **16.** Examine each well for the presence of a precipitate. Indicate which wells contain a precipitate on Template 3.

Hypothesis

Cleanup and Disposal

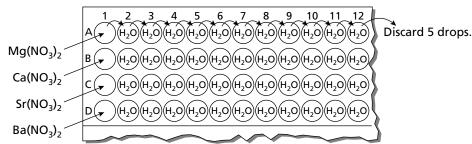
- **1.** Dispose of all chemicals and solutions as directed by your teacher.
- **2.** Return all lab equipment to its proper place.
- **3.** Wash your hands thoroughly with soap or detergent before you leave the lab.

Data and Observations

Use your three templates to record your observations.



Transfer 5 drops from one well to the next.



CHEMISTRY SMALL-SCALE LABORATORY MANUAL

Analyze and Conclude

- **1. Measuring and Using Numbers** Explain how the concentration of alkaline earth metals (amount of metal ion per drop of solution) varied within each row of wells on the microplates.
- **2. Collecting and Interpreting Data** Which alkaline earth metal(s) continued to form precipitates as the concentration became more dilute?
- **3. Observing and Inferring** Which alkaline earth metal(s) did not form precipitates at any concentration?
- **4. Drawing a Conclusion** Compounds with a lower solubility in water will form precipitates in wells with a lower concentration of metal ions. Use this information to describe the general pattern of solubility from magnesium to barium of compounds containing these metals.
- **5. Error Analysis** Compare the results of this experiment with your hypothesis. Explain possible reasons for any disagreement.

Real-World Chemistry

- 1. Water that contains high concentrations of magnesium or calcium ions along with carbonate (CO₃⁻) ions can form deposits that may clog pipes. Based on your observations in this experiment, which metal ion—magnesium or calcium—do you think would contribute more to such deposits if both ions were present in equal concentrations? Explain your reasoning.
- **2.** The alkaline earth metal beryllium exists naturally in compounds that almost always are
- mixed with aluminum compounds. Explain why it is difficult to isolate pure beryllium from these mixtures.
- **3.** Physicians can treat some types of cancer by placing small amounts of a radioactive element in a sealed tube and inserting the tube in the cancerous tissue. Which of the alkaline earth metals could be used for this kind of treatment? Why?



CHEMISTRY SMALL-SCALE LABORATORY MANUAL



Properties of Transition Metals

ike other metals, transition metals are malleable, lustrous, and good conductors of electricity. However, a variety of physical and chemical properties distinguish the transition metals from other metals. There also is considerable variability in the properties of the transition metals themselves. This variability results from differences in their electron configurations. In this activity, you will discover how transition metals differ chemically from other metals.

Problem

How can transition metals be distinguished chemically from other metals?

Objectives

- Observe the physical properties of ten metal ions in aqueous solution.
- Observe the results of mixing three chemicals with each of the solutions.
- Compare the chemical reactivity of transition metal ions with that of other metal ions.

Materials

96-well template $0.1M \text{ Fe}(NO_3)_3$ 96-well microplate $0.1M \operatorname{Co(NO_3)_2}$ thin-stem 0.1M Ni(NO₃)₂ pipettes (15) $0.1M \, Cu(NO_3)_2$ 0.1M KNO₃ $0.1M \, \text{Zn}(NO_3)_2$ $0.1M \, Ca(NO_3)_2$ 6M NH₃ $0.1M \text{ NH}_{A} \text{VO}_{3}$ 1M KSCN $0.1M \text{ Cr(NO}_3)_3$ 6M HCl $0.1M \text{ Mn(NO}_3)_2$ toothpicks (40)

Safety Precautions



- Always wear safety goggles, gloves, and a lab apron.
- Several solutions are poisonous. HCl is corrosive, and HCl and NH₃ will irritate the eyes, skin, and respiratory tract.
- Do not mix HCl and KSCN.
- Use extra care when handling all solutions.
- Notify your teacher of any spills.
- Do not dispose of wastes in the sink or trash can.
- Do not inhale vapors that are released.

Pre-Lab

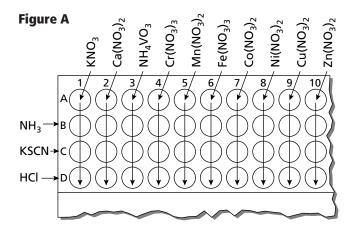
- **1.** What are transition metals?
- **2.** Identify the metals in the ten solutions to be tested in this experiment (K, Ca, V, Cr, Mn, Fe, Co, Ni, Cu, and Zn).
- **3.** Characterize the ten metals as group 1A metals, group 1B metals, or transition metals.
- **4.** Read the entire laboratory activity. Form a hypothesis about which three metal ions will have

properties that are most different from those of the other metal ions. Explain why. Record your hypothesis on page 18.

Procedure

- **1.** Label the 96-well template as shown in **Figure A.**
- **2.** Set the microplate on top of the template with the lettered rows on the left.

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- **6.** Add 5 drops of 1*M* KSCN to each of wells C1–C10. Stir the mixture in each well with a toothpick.
- **7.** Add 5 drops of 6*M* HCl to each of wells D1–D10. Stir the mixture in each well with a toothpick.

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,	PV	•	-	

- **3.** Use a pipette to place 5 drops of 0.1*M* KNO₃ in each of wells A1, B1, C1, and D1. **CAUTION: Never place the pipette in your mouth.**
- **4.** Repeat step 3 for columns 2–10, using the solution assigned to each column on the template. For example, place 5 drops of $0.1M \text{ Ca(NO}_3)_2$ in each of wells A2, B2, C2, and D2.
- **5.** Add 5 drops of 6*M* NH₃ to each of wells B1–B10. Stir the mixture in each well with a toothpick.

Cleanup and Disposal

- **1.** Dispose of all chemicals and solutions as directed by your teacher.
- **2.** Return all lab equipment to its proper place.
- **3.** Wash your hands thoroughly before you leave the lab.

Data and Observations

		Data Table 1			
Color of Effect of adding other chemicals					
Solution	solution	NH ₃	KSCN	HCI	
KNO ₃					
Ca(NO ₃) ₂					
NH ₄ VO ₃					
Cr(NO ₃) ₃					
Mn(NO ₃) ₂					
Fe(NO ₃) ₃					
Co(NO ₃) ₂					
Ni(NO ₃) ₂					
Cu(NO ₃) ₂					
Zn(NO ₃) ₂					

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

- 1. Note the color of each solution in wells A1–A10. Record your observations in **Data Table 1**.
- **2.** For each of wells B1–D10, note whether or not there was a chemical reaction. If there was a reaction that formed a precipitate, indicate the color of the precipitate in **Data Table 1.** If there was a color change without precipitate formation, indicate the new color. If there was no precipitate formation or color change, write *none* in **Data Table 1.**

Analyze and Conclude

1.	Observing and Inferring Which mixtures caused precipitates to form?				
2.	Thinking Critically If no precipitate was formed when a chemical was added to a solution, does that mean a reaction did not occur? Explain.				
3.	Observing and Inferring Which solutions did not appear to react with any of the other chemicals?				
4.	Error Analysis Compare the results of this experiment with your hypothesis. Explain possible reasons for any disagreement.				

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

Real-World Chemistry

- 1. Distillation is one method that can be used to separate a specific metal from a mixture of other elements. Distillation involves raising the temperature of a mixture until one of its components turns into a gas and then cooling the gas to recover that component as a liquid or solid. If there were a mixture containing all of the period 4 transition metals, which metal would be easiest to separate by distillation? Explain your reasoning. (Hint: Use a reference such as the *CRC Handbook of Chemistry and Physics* to find the boiling points of these metals.)
- 2. Cobalt chloride solution can be used as an "invisible ink." The solution leaves no detectable mark when it is applied to paper with a pen. However, heating the paper reveals the message written by the pen. Use your results in this experiment to predict the color of cobalt chloride ink on heated paper.
- **3.** Many transition metals, including vanadium, chromium, manganese, and nickel, are included in alloys that are used to make products such as armor plate, safes, transmission gears, and high-speed metal-cutting tools. Explain why transition metals are used for such alloys.



CHEMISTRY SMALL-SCALE LABORATORY MANUAL



Modeling Molecular Shapes

molecule consists of two or more atoms held together by covalent bonds. The shape of a molecule depends primarily on two factors: the number of covalent bonds formed by each atom, and the number of unshared (lone) pairs of electrons. These two factors are taken into account by the valence shell electron pair repulsion (VSEPR) model, which is used to predict molecular shapes. In this activity, you will model shapes of molecules.

Problem

How can you model the shapes of molecules in the laboratory?

Objectives

- Construct models of molecules by using inflated balloons.
- Observe how varying the number of covalent bonds and lone pairs of electrons affects molecular shape.

Materials

round balloons (4) pear-shaped balloons (6) clear adhesive tape string

Safety Precautions



- Always wear safety goggles and a lab apron.
- Balloons containing latex may cause allergic reactions.

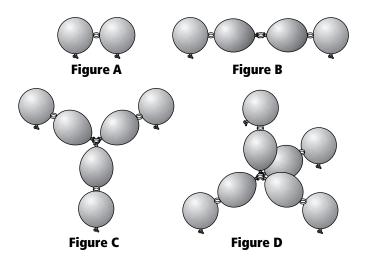
Pre-Lab

- **1.** How does a covalent bond differ from an ionic bond?
- **2.** How does the VSEPR model take into account the repulsion of electron pairs in predicting molecular shape?
- **3.** What effect do lone pairs of electrons have on shared bonding orbitals? Why?
- **4.** What is hybridization?
- **5.** Read the entire laboratory activity. Form a hypothesis about the shapes of molecules made of 2, 3, 4, and 5 atoms with no lone pairs of electrons. Record your hypothesis on page 22.

Procedure

- **1.** Inflate the round balloons to the same size and tie their ends closed.
- **2.** Inflate four pear-shaped balloons to the same size and tie their ends closed.
- **3.** For each balloon, make a loop of tape by wrapping the tape around two fingers. The adhesive side of the tape should face out.
- **4.** Attach one tape loop to each balloon. On the pear-shaped balloons, put the tape on the end opposite where the knot is tied.
- 5. Push two of the round balloons together so that the tape loop on each balloon sticks to the other balloon, as shown in Figure A. This is a model of the hydrogen molecule, H₂. Describe the shape of the model in Data Table 1.

CHEMISTRY SMALL-SCALE LABORATORY MANUAL



- **6.** Disassemble the H_2 model.
- 7. Use string to tie together the knotted ends of two pear-shaped balloons. Attach a round balloon to the other end of each pear-shaped balloon, as shown in Figure B. This is a model of beryllium dichloride, BeCl₂. Describe the shape of the model in Data Table 1.
- **8.** Use string to tie a third pear-shaped balloon to the knotted ends of the two pear-shaped balloons in the BeCl₂ model. Attach a round balloon to the other end of the third pear-shaped balloon. The resulting structure, which should resemble **Figure C**, is a model of aluminum trichloride, AlCl₃. Describe the shape of the model in **Data Table 1**.
- **9.** Use string to tie a fourth pear-shaped balloon to the knotted ends of the three pear-shaped balloons in the AlCl₃ model. Attach a round balloon to the other end of the fourth pear-shaped balloon. The resulting structure, which should resemble **Figure D**, is a model of methane, CH₄. Describe the shape of the model in **Data Table 1**.
- **10.** Remove two of the pear-shaped balloons and their attached round balloons from the CH₄ model.

- **11.** Inflate two pear-shaped balloons so that they are at least twice the size of the other pear-shaped balloons. Tie their ends closed. Each large pear-shaped balloon represents a lone pair of electrons.
- **12.** Use string to tie the large pear-shaped balloons to the knotted ends of the two remaining pear-shaped balloons in the partially disassembled CH₄ model. The resulting structure is a model of water, H₂O. Describe the shape of the model in **Data Table 1.**

Hypothesis

Cleanup and Disposal

- **1.** Return all lab equipment to its proper place.
- **2.** Wash your hands thoroughly with soap or detergent before you leave the lab.

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

Data and Observations

	Data Table 1			
Molecule	ule Shape Bond angle (°)			
H ₂				
BeCl ₂				
AICI ₃				
CH ₄				
H ₂ O				

Calculate the bond angles in each of the models you constructed. Record the bond angles in **Data Table 1.**

Analyze and Conclude

- **1. Formulating Models** What type of orbital is represented by a round balloon? A pear-shaped balloon? (Hint: Think about the shape of each balloon.)
- **2. Applying Concepts** What hybrid orbitals are formed in each of the molecules you modeled?
- **3. Observing and Inferring** Both BeCl₂ and H₂O contain three atoms. Do your models show that these molecules have the same shape? Explain why or why not.
- **4. Predicting** Predict the shapes of the following molecules: CH₃Cl, Cl₂, CCl₄, HCl, BF₃.

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5.	Error Analysis Compare the results of this experiment with the predictions of your
	hypothesis. Explain possible reasons for any disagreement.

Real-World Chemistry

- Ethene, H₂C = CH₂, serves as the starting material for the synthesis of polyethylene, from which plastic bags and milk jugs are made. Ethyne, H—C≡C—H, is used as a fuel for welding torches. The double bond in ethene and the triple bond in ethyne have the same effect on molecular shape as single bonds. Predict the shapes and bond angles of ethene and ethyne.
- 2. Hemoglobin is an iron-containing molecule that transports oxygen in your blood. Each iron atom in hemoglobin forms bonds with five nitrogen atoms and one oxygen atom. Use the VSEPR model to predict the shape of the part
- of hemoglobin that consists of iron and the atoms it bonds with. (Hint: Count the number of shared electron pairs involved and refer to Table 9-3 in your textbook.)
- **3.** Living things are made of a huge variety of different carbon-containing molecules. Many of these molecules are very large and have complex, three-dimensional shapes. Would the same variety of shapes be possible for molecules that contain beryllium instead of carbon? Explain why or why not. (Hint: Remember that carbon atoms can form bonds with other carbon atoms.)

CHEMISTRY SMALL-SCALE LABORATORY MANUAL



Class

Solutions and Precipitates

queous solutions of ionic compounds contain dissolved positive and negative ions. When two such solutions are mixed, the ions may take part in a double-replacement reaction. One outcome of a double-replacement reaction is the formation of a precipitate. By writing ionic equations and knowing the solubilities of specific ionic compounds, you can predict whether a precipitate will be formed.

Problem

Can you predict whether two aqueous solutions will form a precipitate when they are mixed?

Objectives

- Write ionic equations for mixtures of aqueous solutions.
- Predict which mixtures will form precipitates.
- **Observe** mixtures for precipitate formation.

Materials

1.0*M* BaCl₂ 1.0*M* NaOH 1.0*M* CuSO₄ 1.0*M* Na₂SO₄ 1.0*M* FeCl₃ 1.0*M* Pb(NO₃)₂ 1.0*M* KI 24-well microplate 1.0*M* NaCl pipettes (9) 1.0*M* Na₂CO₃

Safety Precautions



- Always wear safety goggles, gloves, and a lab apron.
- Use extra care when handling the solutions.
- Notify your teacher of any spills.
- Do not dispose of wastes in the sink or trash can.
- Never place the pipette in your mouth.

Pre-Lab

- **1.** What is a double-replacement reaction?
- **2.** What are the three types of products that can form from double-replacement reactions?
- **3.** What is a spectator ion?
- **4.** What is the difference between a complete ionic equation and a net ionic equation?
- **5.** Read the entire laboratory activity. Study **Table 1.** Form a hypothesis about which mixtures listed in **Data Table 1** will form a precipitate. Explain why a precipitate will form in those mixtures. Record your hypothesis on page 26.

Table 1

Solubility of Ionic Compounds in Water							
Anion							
CI-	CI						
S	I	1	S	Р	I		
S	I	S	S	I	S		
S	_	S	S	ı	S		
S	S	S	S	S	S		
S	S	S	S	S	S		
Р	I	Р	S	ı	ı		
	ci - s s s s	CI ⁻ CO ₃ ²⁻ S I S I S - S S S S	CI	Anion Cl⁻ CO₃²⁻ I⁻ NO₃⁻ S I I S S I S S S — S S S S S S S S S S S S S S	Anion Cl⁻ CO₃²⁻ I⁻ NO₃⁻ OH⁻ S I I S P S I S S I S — S S I S S S S S S S S S S S S S S S		

Key

I—insoluble P—partially soluble

S—soluble

LAB (7)

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

Procedure

- **1.** Choose any one of the mixtures listed in **Data Table 1.** Use a pipette to add 5 mL of the first solution in that mixture to a well on the microplate.
- **2.** Use a different pipette to add 5 mL of the second solution in the mixture to the same well. Do not allow the tip of the pipette to touch the mixture.
- **3.** Record the number and letter of the well in **Data Table 1.**
- **4.** Repeat steps 1–3 for the other ten mixtures listed in **Data Table 1.** Use a different well for each mixture.

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Cleanup and Disposal

- **1.** Dispose of all chemicals and solutions as directed by your teacher.
- **2.** Return all lab equipment to its proper place.
- **3.** Wash your hands thoroughly before you leave the lab.

Data and Observations

Data Table 1				
Mixture	Solutions	Well	Observations	
1	CuSO ₄ and NaOH			
2	FeCl ₃ and NaOH			
3	KI and NaOH			
4	KI and NaCl			
5	KI and Pb(NO ₃) ₂			
6	Na ₂ SO ₄ and Pb(NO ₃) ₂			
7	Na ₂ SO ₄ and BaCl ₂			
8	Na ₂ CO ₃ and BaCl ₂			
9	Na ₂ CO ₃ and KI			
10	Na ₂ CO ₃ and CuSO ₄			
11	NaCl and CuSO ₄			

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

- **1.** Carefully observe each well that contains a mixture of solutions and note whether or not a precipitate is visible. Record the results in **Data Table 1.**
- 2. If there are any other signs that a reaction has occurred in any of the wells, describe those signs in **Data Table 1.**

Analyze and Conclude

1.	Observing and Inferring Write the numbers of the mixtures that resulted in a double-replacement reaction. Explain how you know a reaction occurred in those mixtures.
2.	Applying Concepts Write the complete ionic equations of the reactions that occurred.
3.	Thinking Critically Why is it impossible to write a net ionic equation for any of the mixtures that did not form a precipitate?
4.	Error Analysis Compare the results of this lab with the predictions of your hypothesis. Explain possible reasons for any disagreement.

LAB (7)

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

Real-World Chemistry

- **1.** Seawater is a dilute solution of several ionic compounds, the major one of which is sodium chloride (NaCl). One way to measure the amount of NaCl in a sample of seawater is to mix the sample with a solution of silver nitrate (AgNO₃). Explain how this method is likely to work. (Hint: AgCl is insoluble in water.)
- 2. Cells that line your stomach secrete hydrochloric acid (HCl), which helps you digest your food. When these cells secrete too much HCl, an upset stomach may result. To relieve an upset stomach, you may take an
- antacid, such as magnesium hydroxide, Mg(OH)₂. Magnesium hydroxide reacts with HCl in a double-replacement reaction, but no precipitate is formed. Write the complete and net ionic equations for this reaction. What is the product?
- **3.** Sodium hydrogen carbonate (NaHCO₃) can also be used as an antacid. Write the net ionic equation for the reaction between NaHCO₃ and HCl. What would be one disadvantage of using NaHCO₃ instead of Mg(OH)₂ to treat an upset stomach?

CHEMISTRY SMALL-SCALE LABORATORY MANUAL



Determining Avogadro's Number

vogadro hypothesized that equal numbers of moles of different gases at the same temperature and pressure contain the same number of molecules. But he never calculated what that number might be. Later on, other scientists calculated a value for Avogadro's number. In one experiment, a thin film of a chemical was spread on the surface of water. The layer was assumed to be one molecule thick, or a monolayer. In 1924, it was estimated that the number of molecules in a mole was 6.004×10^{23} .

You will cover the surface of a water sample with a monolayer of stearic acid by adding drops of stearic acid solution to the water surface. The solvent will quickly evaporate, leaving the nonpolar stearic acid molecules on the water's surface.

The mass of the stearic acid can be determined, and the number of moles of stearic acid can be calculated using the molar mass of stearic acid. The monolayer formed is one molecule thick, so if an assumption is made about the shape of a single molecule, the number of molecules in the monolayer can be estimated. Avogadro's number is then estimated as the ratio of the number of molecules of stearic acid to the number of moles of stearic acid. The closer the assumed shape is to the actual shape of the molecule, the more precise the calculation of Avogadro's number will be. In this experiment, the shape of the molecule is first assumed to be a rectangular solid and then a cylindrical solid.

Problem

How many molecules of stearic acid are in a mole of stearic acid?

Objectives

- Measure the diameter of stearic acid solution in a monolayer.
- Calculate a value for Avogadro's number.
- Infer which volume estimate better approximates the volume of a stearic acid molecule.

Materials

stearic acid solution distilled water lycopodium powder or talcum powder 50-mL beaker 10-mL graduated cylinder metric ruler iron ring
Pasteur pipette
with suction bulb
large watch glass
ring stand
small test tubes (2)
test-tube rack
scoop

Safety Precautions



- Always wear safety goggles, gloves, and a lab apron.
- Avoid breathing directly over the watch glass.

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

Pre-Lab

- **1.** What is the accepted value for Avogadro's number?
- **2.** Calculate the volume of a cylinder that has a diameter of 3.00×10^{-4} cm and a height of 1.00×10^{-2} cm. $(V_{\rm cyl} = \pi (d/2)^2 \times h)$
- **3.** Stearic acid is a solid at room temperature and pressure. For this experiment, it must be dissolved in an appropriate solvent. Why must the chosen solvent evaporate quickly from the water's surface?
- **4.** Read the entire laboratory activity. Two assumptions are made about the shape of a single stearic acid molecule. Form a hypothesis as to which shape you think will give the more precise value for Avogadro's number—rectangular solid or cylindrical solid. Record your hypothesis in the next column.

Procedure

- Fill a 50-mL beaker with distilled water. Attach a suction bulb to a Pasteur pipette and fill it with distilled water. Count the number of drops needed to fill a 10-mL graduated cylinder to the 1.00 mL mark. Record the number of drops in Data Table 1. Repeat this process two more times.
- **2.** Set the watch glass on a metal ring, which is attached to a ring stand. Place the ring at a level so that the top of the watch glass can be viewed at eye level.
- 3. Using a ruler, measure and record the diameter of the watch glass. Thoroughly wash the watch glass and rinse it several times with distilled water. CAUTION: All soap must be rinsed off or a monolayer of molecules will not form. Replace the watch glass on the metal ring and fill it with distilled water.
- **4.** Pour 2 mL of stearic acid solution into a small test tube. Set the test tube in a test-tube rack. Label a second small test tube "WASTE" (material can be recycled) and set it in the test-tube rack.

- **5.** Fill the end of a scoop with lycopodium powder and, while holding the scoop about 30 cm above the watch glass, uniformly sprinkle the powder over the surface of the water. The powder layer should be thin—like a layer of dust.
- **6.** Rinse the Pasteur pipette with a small amount of the stearic acid solution. Put the rinsing into the test tube labeled WASTE (material can be recycled). Partially fill the Pasteur pipette with half of the remaining solution.
- **7.** While holding the pipette directly above the center of the watch glass, squeeze out one drop of the solution.
- **8.** Observe the drop as it spreads over the surface of the water in the watch glass. When the drop has finished spreading, add a second drop. The amount of spreading will decrease with each additional drop. Continue to add drops one at a time until the added drop does not spread. Record the total number of drops in **Data Table 2.**
- **9.** Empty and clean the watch glass, making sure that all of the soap is removed.
- **10.** Repeat steps 3 and 5. Fill the Pasteur pipette with the second half of the stearic acid solution. Repeat steps 7 and 8.

Hypothesis

Cleanup and Disposal

- 1. Wash, dry, and store all equipment
- **2.** Dispose of the stearic acid solution in the waste test tube in the designated container.
- **3.** Wash your hands thoroughly with soap or detergent before you leave the lab.

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

Data and Observations

	Data Tab	le 1		
	Calibration o	of pipette		
	Trial 1	Trial 2	Trail 3	Average
Number of drops in 1.00 mL				

	Data Table 2	
	Trial 1	Trial 2
Drops of stearic acid solution needed to form monolayer		

Density of stearic acid: 0.9405 g/cm³

Concentration of stearic acid solution: 0.10 g/L

Molar mass of stearic acid: 284.5 g/mol

Diameter of the watch glass (cm):

- 1. Calculate the average number of drops in one milliliter for your Pasteur pipette.
- **2.** Use the following equation to calculate the mass of stearic acid.

Mass of stearic acid = Concentration (g/L) \times 1 L/1000 mL \times 1 mL/avg. no. of drops \times no. of drops to form monolayer

- **3.** Calculate the volume of the monolayer by dividing the mass of stearic acid (step 2) by the density of stearic acid.
- **4.** The watch glass is circular, so the monolayer of stearic acid covering its surface is a cylinder. To calculate the thickness of the monolayer, divide the volume of the monolayer (step 3) by the area of the circle. [$Area = \pi(diameter(cm)/2)^2$]
- **5.** The monolayer is one molecule thick, so the thickness of a stearic acid molecule is equal to the length of the molecule. If the length of the molecule is assumed to be six times the width and depth, calculate the volume of one molecule when the shape of the stearic acid molecule is assumed to be a rectangular solid. $(V_{rec} = l \times w \times d)$; (l = 6w = 6d)
- **6.** Find the number of molecules by dividing the volume of the monolayer (step 3) by the volume of one molecule (step 5).

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

- **7.** Calculate the number of moles of stearic acid in the monolayer by dividing the mass of stearic acid (step 2) by the molar mass of stearic acid.
- **8.** To calculate Avogadro's number when the stearic acid molecule is a rectangular solid, divide the number of molecules (step 6) by the number of moles (step 7).
- **9.** If the length of the molecule is assumed to be six times the diameter, calculate the volume of one molecule when the shape of the stearic acid molecule is assumed to be a cylindrical solid. $(V_{\text{cyl}} = l \times (\pi(d/2)^2); (l = 6d)$
- **10.** To find the number of molecules, divide the volume of the monolayer (step 3) by the volume of one molecule (step 9).
- **11.** Recalculate Avogadro's number, assuming the stearic acid molecule is a cylindrical solid. Divide the number of molecules (step 10) by the number of moles (step 7).

Analyze and Conclude

- **1. Error Analysis** The accepted value for Avogadro's number is 6.022×10^{23} particles per mole. Calculate the percent error for each of your calculations of Avogadro's number.
- **2. Drawing a Conclusion** Which of your two calculations came closer to the accepted value for Avogadro's number—the calculation made assuming a rectangular solid or the calculation made assuming a cylindrical solid?
- **3. Observing and Inferring** Which assumption is closer to the actual shape of the stearic acid molecule—rectangular solid or cylindrical solid?

Real-World Chemistry

To calculate Avogadro's number, chemists made assumptions about the shape of a single molecule. Chemists who use computer simulations to model compounds and chemical reactions also make simplifying assumptions about molecular shape

in order to perform calculations needed for their research. Identify and investigate a program used by chemists for computer simulation and list some of the limitations and assumptions this program makes.

Class



CHEMISTRY SMALL-SCALE



Measuring Boiling Point

■aporization is the phase change in which a liquid changes to a gas. When vaporization occurs throughout a liquid instead of only at the surface, the liquid is said to be boiling. The temperature at which boiling occurs, called the boiling point, varies for different substances. Two factors—molar mass and the ability to form hydrogen bonds—are most important in determining boiling point. In general, boiling points are higher for more massive molecules and for molecules that form hydrogen bonds.

Problem

How can you compare the boiling points of substances?

Objectives

- Measure the boiling points of three known liquids.
- Correlate boiling point with molar mass and the ability to form hydrogen bonds.
- **Identify** an unknown liquid by its boiling point.

Materials

acetone ethanol 1-propanol 100-mL beaker thermometer $(-10^{\circ}\text{C to }100^{\circ}\text{C})$ capillary tubes, sealed at one end (3)

small test tubes (3) thin-stem pipette hot plate ring stand thermometer clamp rubber band glass stirring rod

Safety Precautions













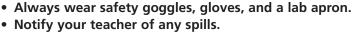












- Use caution and proper protection when handling hot objects.
- Do not dispose of wastes in the sink or trash can.
- Never place the pipette in your mouth.
- Acetone, ethanol, and propanol are all flammable chemicals.

Pre-Lab

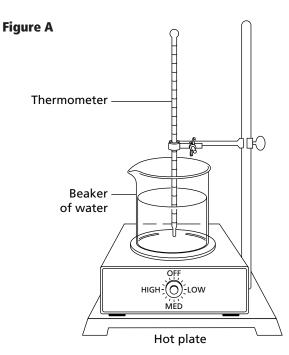
- **1.** Contrast evaporation and boiling.
- **2.** Define *vapor pressure*.
- **3.** What is the relationship between vapor pressure and external pressure when a liquid is at its boiling point?
- **4.** What are the signs that a liquid has reached its boiling point?
- **5.** Read the entire laboratory activity. Form a hypothesis about which of the substances you will test has the highest boiling point and which has the lowest boiling point. Explain your reasoning. Record your hypothesis on page 34.

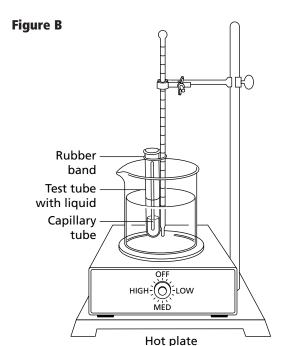
Procedure

- **1.** Set the hot plate on the base of the ring stand.
- **2.** Fill the beaker about two-thirds full with tap water. Place the beaker on the hot plate.
- **3.** Use the thermometer clamp to attach the thermometer to the ring stand. Lower the clamp so the thermometer is in the water, as shown in **Figure A** on page 34.
- **4.** Turn the hot plate on to a medium setting. While the water is heating, complete steps 5 and 6.
- **5.** Place a clean capillary tube with the sealed end up into a clean, small test tube.

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- **6.** Use the pipette to add 3 mL of one of the known liquids to the test tube.
- **7.** When the water temperature has reached approximately 80°C, raise the clamp so the thermometer is above the beaker. **CAUTION:** The thermometer will be hot.
- **8.** Use the rubber band to attach the test tube to the thermometer. The bottom of the test tube should be at the same level as the bulb of the thermometer.
- **9.** Lower the clamp so the test tube—thermometer assembly is in the water. The top of the assembly should be above the surface of the water, as shown in **Figure B.**
- **10.** Look for bubbles coming from the open end of the capillary tube. If you do not see any bubbles, continue heating the water until you do.
- **11.** When a steady stream of bubbles issues from the open end of the capillary tube, turn off the hot plate.
- 12. Use the glass stirring rod to stir the water constantly until the stream of bubbles stops. Immediately read the temperature of the water to the nearest 0.1°C. This is the boiling point of the liquid in the capillary tube. Record the temperature in **Data Table 1.**





13. Repeat steps 4–12 with the other two known liquids and the unknown liquid. Use distilled water to clean the pipettes before drawing up a different liquid.

Hypothesis

Cleanup and Disposal

- **1.** Dispose of all materials as directed by your teacher.
- **2.** Return all lab equipment to its proper place.
- **3.** Wash your hands thoroughly with soap or detergent before you leave the lab.

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

Data and Observations

		Data Table 1		
Liquid	Molar formula	Molar mass (g)	Hydrogen bonding?	Boiling point (°C)
Acetone	C ₃ H ₆ O		no	
Ethanol	C ₂ H ₅ O		yes	
1-propanol	C ₃ H ₈ O		yes	
Unknown	_	_	_	

Analyze and Conclude

- **1. Measuring and Using Numbers** Calculate the molar mass of each known liquid. Record the results in **Data Table 1.**
- **2. Thinking Critically** Why did you record the temperature when the bubbles stopped appearing as the boiling point of each liquid?
- **3. Observing and Inferring** Identify the unknown liquid by comparing its boiling point with the boiling points of the known liquids.
- **4. Drawing a Conclusion** Based on your results, which factor—molar mass or hydrogen bonding—seems to be more important in determining boiling point? Explain your reasoning.

5. Acquiring and Analyzing Information Look up the accepted boiling point of each known liquid.

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

6.	Error Analysis Compare the boiling point you measured for each known liquid with
	the accepted boiling point. Calculate the percent error, if any. Explain possible sources of
	error in the lab.

Real-World Chemistry

- **1.** The amount of time it takes to cook noodles depends on the temperature of the water that they are cooked in. Would it take longer to cook noodles in boiling water at sea level or at an altitude of 3000 m? Explain.
- **2.** Why are foods such as french fries and fried chicken cooked in oil rather than in water? (Hint: Cooking oil is a mixture of fat molecules whose molar masses may be 800 g or more.)
- **3.** Most automobile engines are cooled by a mixture of water and antifreeze, which consists mostly of 1,2-ethanediol (commonly called ethylene glycol). Why is such a mixture better at cooling an engine than water alone? (Hint: The boiling point of 1,2-ethanediol is 197°C.)

(10)

CHEMISTRY SMALL-SCALE LABORATORY MANUAL



Relating Gas Pressure and Gas Volume

pressure, volume, and temperature—that determine the behavior of gases. One of the gas laws, Boyle's law, states how gas pressure and gas volume are related. Boyle's law applies to ideal gases, which obey all of the assumptions made by the kinetic theory. With a simple laboratory setup, you can test how well Boyle's law applies to real gases.

Problem

How does changing the pressure of a gas affect the volume of the gas?

Objectives

- Measure the volume of the air sample at different pressures.
- Make and use a graph of volume versus pressure to illustrate the mathematical relationship.

Materials

flat objects that can be stacked (4) Boyle's law apparatus carpet thread balance metric ruler

Safety Precautions



- Always wear safety goggles, gloves, and a lab apron.
- Gas under pressure can be hazardous. Use the Boyle's law apparatus only as directed.

Pre-Lab

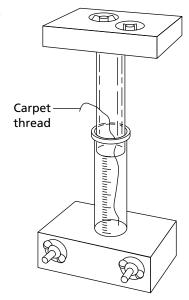
- **1.** State Boyle's law.
- **2.** Define pressure.
- **3.** A force of 12.7 N is applied to a circular disk whose radius is 6.20 cm. Calculate the pressure (in N/m²) applied to the disk. (Hint: The area of a circle = $\pi \times \text{radius}^2$.)
- **4.** Suppose there is an inverse relationship between two variables, *x* and *y*. What happens to *y* as *x* increases?
- **5.** Read the entire laboratory activity. Form a hypothesis about how the volume of the air sample will change as the pressure on the sample increases. Record your hypothesis on page 38.

Procedure

- **1.** Use the balance to measure the mass of each object to the nearest gram. Record the masses in **Data Table 1.**
- **2.** Pull the plunger all the way out of the Boyle's law apparatus. Use the ruler to measure the diameter of the head of the plunger to the nearest millimeter. Record the diameter in **Data Table 1.**
- **3.** Insert a length of carpet thread into the barrel of the Boyle's law apparatus. Leave one end of the thread hanging out of the apparatus, as shown in **Figure A.**

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

Figure A



- **4.** Push the plunger back into the barrel until the head of the plunger is near the 35 cm³ mark on the barrel. The thread will make a small space for air to escape past the head as you do this.
- **5.** While holding the plunger in place, carefully remove the thread. Twist the plunger several times to reduce friction between the head and the barrel.
- **6.** Measure the volume to the nearest 0.1 cm³. Record the volume in **Data Table 1** as the volume for zero total mass under *Trial 1*.

- **7.** Carefully place the first mass on top of the plunger.
- **8.** While stabilizing the apparatus, twist the plunger several times to reduce friction. Measure and record the volume to the nearest 0.1 cm³ under *Trial 1*.
- **9.** Carefully stack the second mass on top of the first mass. Repeat step 8.
- **10.** Carefully stack the third mass on top of the other two masses. Repeat step 8.
- **11.** Carefully stack the fourth mass on top of the other three masses. Repeat step 8.
- **12.** Remove all four masses. Repeat steps 6–11 two more times. Record the results in **Data Table 1** under *Trial 2* and *Trial 3*.

Hypothesis

Cleanup and Disposal

- **1.** Make sure your balance is left in the same condition as you found it.
- **2.** Return all lab equipment to its proper place.

Data and Observations

Mass of ob	ject 1 (g):				Mas	s of object 3	(g):		
Mass of ob	oject 2 (g):				Mas	s of object 4	(g):		
Diameter of	of plunger he	ad (mm):			Are	a of plunger	head (m²):		
Stacked	Total		Pressure	Tot			Volum	e (cm³)	
objects	mass (kg)	Force (N)	(N/m ²)	(N/r	_	Trial 1	Trial 2	Trial 3	Average
none	0								
1									
1, 2									
1, 2, 3									
1, 2, 3, 4									

Data Table 1

- **1.** Calculate the cross-sectional area (in m²) of the head of the plunger. (Remember that radius = diameter/2.) Record the result in **Data Table 1.**
- **2.** Calculate the total mass (in kg) when one, two, three, or all four objects were placed on the apparatus. Record the results in **Data Table 1.**
- **3.** Calculate the force (in N) exerted on the plunger by each total mass. Use the following formula: force (N) = mass (kg) \times 9.80 m/s². Record the results in **Data Table 1.**
- **4.** Calculate the pressure (in N/m²) exerted on the plunger by each total mass. (Hint: Recall the definition of pressure.) Record the results in **Data Table 1.**
- **5.** The atmosphere also exerts pressure on the plunger. For each total mass, calculate the total pressure by adding atmospheric pressure to the pressure exerted by stacked objects. (Unless your teacher instructs you otherwise, assume that atmospheric pressure = 1.0 × 10⁵ N/m².) Record the results in **Data Table 1.**
- **6.** For each total mass, calculate the average volume by adding the volumes in each trial and dividing by 3. Record the results in **Data Table 1.**

Analyze and Conclude

1. Making and Using Graphs Make a graph of volume versus pressure. Plot the average volume on the vertical axis and the total pressure on the horizontal axis. Draw lines between the data points, label both axes, and give the graph a title.

2. Collecting and Interpreting Data Describe the relationship between average volume

and total pressure as shown by the results in Data Table 1 and on your graph.
3. Applying Concepts Would your results have been different if the temperature of the apparatus had changed during the experiment? Explain.
4. Error Analysis Compare the results of this experiment with your hypothesis. Explain possible reasons for any disagreement.

LAB (10)

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

Real-World Chemistry

- **1.** Most gasoline engines have a set of pistons that move up and down inside hollow cylinders. Each up-and-down movement is called a stroke. In the compression stroke, the pistons reduce the volume (compress) a gaseous mixture of fuel and air in the cylinders. A typical gasoline engine in a modern automobile may have a compression ratio of 8 to 1. This means that the volume of gas in each cylinder at the start of the compression stroke is 8 times the volume at the end of the stroke. If the gas is at atmospheric pressure $(1.0 \times 10^5 \text{ N/m}^2)$ at the start of a compression stroke, what is the pressure of the gas at the end of the stroke? Assume the temperature of the gas does not change during the stroke.
- 2. The helium in a weather balloon occupies a volume of 200.0 L when the balloon is released at sea level, where the atmospheric pressure is 1.0×10^5 N/m². What is the volume of the helium when the balloon is at an altitude of 10700 m, where the surrounding air pressure is 2.3×10^4 N/m²? Assume the temperature is the same at the two locations.
- **3.** As a scuba diver descends through the water, the surrounding water pressure increases. The gases in the diver's body, including any small bubbles that may be in the blood vessels, are at the same pressure as the surrounding water. What happens to these bubbles as the diver ascends? Why is it dangerous for a diver to ascend too quickly?

Class



CHEMISTRY SMALL-SCALE



Effect of Temperature on Solubility

er a solute to dissolve in a solvent, the solute and solvent particles must collide with each other. Any factor that affects the frequency of these collisions is likely to affect the rate at which the solute dissolves. One important factor is temperature. As temperature increases, the average kinetic energy of solute and solvent particles increases. Collisions occur more frequently and with greater energy. Therefore, many solutes dissolve more rapidly at higher temperatures. For other solutes, however, the opposite is true. In this activity, you will determine the effects of temperature on the solution process.

Problem

How can you determine the effect of temperature on the rate at which ammonia gas dissolves in water?

Objectives

- Collect ammonia gas from a concentrated ammonia solution.
- **Measure** the time required for ammonia gas to dissolve in water at four different temperatures.
- Relate the time it takes the ammonia to dissolve to solubility.

Materials

concentrated ammonia solution 400-mL beaker 250-mL beaker 100-mL beaker thin-stem pipette

wide-stem pipette scissors metric ruler stopwatch crushed ice fume hood

Safety Precautions



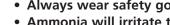












- Always wear safety goggles, gloves, and a lab apron.
- · Ammonia will irritate the eyes, skin, and respiratory tract. Use extra care when handling the concentrated ammonia solution.
- Notify your teacher of any spills.
- Use caution and proper protection when handling hot objects.
- Do not dispose of wastes in the sink or trash can.

Pre-Lab

- **1.** What is solubility?
- **2.** Refer to Table 15-2 in your textbook. How does decreasing the temperature affect the solubility of most solids that are dissolved in water?
- **3.** How does decreasing the temperature affect the solubility of gases in liquid solvents?
- **4.** How would the solubility of a gas in a liquid change if the pressure of the gas above the liquid was doubled? Explain why.
- **5.** Read the entire laboratory activity. Form a hypothesis about how decreasing the temperature will affect the time required for ammonia to dissolve in water. Explain why decreasing the temperature should have this effect. Record your hypothesis on page 42.

LAB (11)

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

Procedure

- **1.** Use scissors to cut the tip off the wide-stem pipette, leaving the stem about 4 cm long. This is the collecting pipette.
- **2.** Place the three beakers used in this activity under the fume hood. Beakers must remain beneath the fume hood throughout the entire procedure. Fill the 400-mL beaker with about 350 mL of hot tap water.
- **3.** Working under the fume hood, halfway fill the thin-stem pipette with concentrated ammonia solution. This is the generating pipette. Keep the pipette in the fume hood at all times.
- **4.** Turn the generating pipette bulb-end down and submerge the bulb in the beaker of hot water. Cover the tip of the generating pipette with the collecting pipette, as shown in **Figure A.**
- **5.** As the ammonia solution warms in the generating pipette, ammonia gas leaves the solution and begins filling the bulb of the collecting pipette. Squeeze the collecting pipette several times to expel all of the air and help the ammonia gas completely fill the collecting pipette.

Figure A

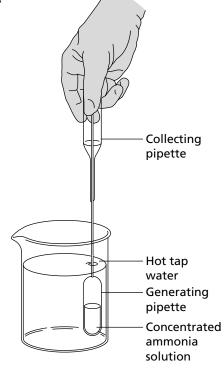
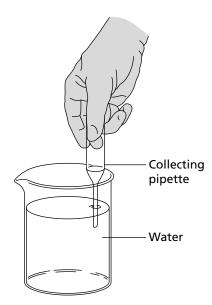


Figure B



- **6.** Fill the 250-mL beaker with 200 mL of hot tap water. Measure the temperature of the water to the nearest 0.1°C. Record the temperature in **Data Table 1.**
- **7.** After collecting ammonia gas for 3–4 min, remove the collecting pipette from the generating pipette. Set the generating pipette bulb down in the 100-mL beaker.
- **8.** Submerge the tip of the collecting pipette in the hot water in the 250-mL beaker, as shown in **Figure B.** Start the stopwatch. Keep the level of water in the pipette near the level in the beaker by lowering the pipette as it fills with water.
- **9.** Stop the stopwatch when the gas in the collecting pipette has dissolved completely. The gas is completely dissolved when the pipette is full of water. Record the time in **Data Table 1.**
- **10.** Repeat steps 2 through 9, filling the 250-mL beaker with 200 mL of each of the following.
 - **a.** a 50-50 mixture of hot tap water and cold tap water
 - **b.** cold tap water
 - **c.** a mixture of cold tap water and ice

LAB	(11)

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

Hypothesis	Cleanup and Disposal
	1. Dispose of all solutions as directed by your teacher.
	2. Return all lab equipment to its proper place.
	Wash your hands thoroughly with soap or detergent before leaving the lab.

Data and Observations

	Data 1	Table 1	
	Temperature (°C)	Temperature (K)	Time (s)
Hot water			
Hot water + cold water			
Cold water			
Cold water + ice			

Convert each temperature in degrees Celsius to kelvin by adding 273. Record the results in **Data Table 1.**

Analyze and Conclude

onsider the density of	f the gases involved.)
Applying Concepts	Explain why water was drawn into the collecting pipette in step 8.

3. Making and Using Graphs Make a graph of dissolving time (in seconds) versus temperature (in kelvins). Connect the data points, label the axes, and title the graph.

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

4. Observing and Inferring Based on your results, what can you infer about the effect of temperature on the solubility of ammonia in water? Compare this conclusion with the data given for ammonia in Table 15-2 in your textbook.
5. Error Analysis How do your results compare with the predictions of your hypothesis? Provide an explanation for any disagreement.

Real-World Chemistry

- **1.** Much of the carbon dioxide produced by living things and by the burning of fossil fuels is dissolved in ocean water. Explain how the level of carbon dioxide in the atmosphere would be affected if ocean temperatures were to increase.
- **2.** Some fish that live in stagnant pools come to the surface occasionally to gulp air. Such fish will gulp air more frequently if the water in the pools is warm. Explain why.
- **3.** Is a bottle of carbonated beverage at room temperature more likely to bubble over when opened than one that has been refrigerated overnight? Explain.



CHEMISTRY SMALL-SCALE LABORATORY MANUAL



Specific Heat of Metals

eat flows from a warmer object to a cooler object. As heat flows, the temperature of the warmer object decreases and the temperature of the cooler object increases. The magnitude of the temperature change depends in part upon what each object is made of. Objects that experience a large temperature change when they absorb or release a given amount of heat have a low specific heat. Objects that experience a small temperature change for a given amount of heat transfer have a high specific heat.

Problem

How can you use water to measure the specific heat of metals?

Objectives

- Construct a calorimeter.
- Measure changes in the temperature of water in the calorimeter when warmer metals are added.
- Calculate the specific heat of each metal.

Materials

aluminum, iron, and lead samples plastic-foam cups (2) 400-mL beakers (2) 100-mL graduated cylinder

large test tubes (3) boiling chips scissors balance hot plate thermometer test-tube rack

Safety Precautions



- Always wear safety goggles, gloves, and a lab apron.
- Broken glassware may have sharp edges. Be careful when using scissors.
- Use caution and proper protection when handling hot objects.
- Avoid spilling water on the hot plate power cord.
- Wipe up any water spills immediately to avoid slipping.

Pre-Lab

- **1.** Define specific heat.
- **2.** A sample of substance X has a mass of 123 g. When the sample releases 795 J of heat, its temperature falls from 45.1°C to 17.6°C. What is the specific heat of substance X?
- **3.** What is a calorimeter?
- **4.** Why is it important that a calorimeter be made of an insulating material?
- **5.** Read the entire laboratory activity. Form a hypothesis about which of the three metals will cause the largest change in water temperature for its mass. Explain your reasoning. Record your hypothesis on page 46.

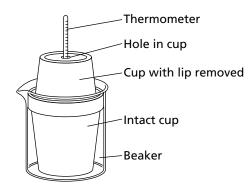
Procedure

- 1. Use scissors to remove the lip from one of the plastic-foam cups. As shown in **Figure A**, this cup will be the top of the calorimeter. Invert it and set it on top of the other cup, which will be the bottom of the calorimeter.
- **2.** Use a pencil to punch a hole in the center of the top of the calorimeter. The hole should be large enough to hold the thermometer.
- **3.** Place the calorimeter in one of the beakers to keep it from tipping over.
- **4.** Measure the mass of each metal sample to the nearest 0.1 g. Record the masses in **Data Table 1.** Place each metal in a separate test tube, and label each tube.

LAB (12)

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

Figure A

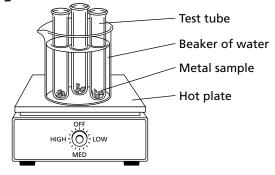


- **5.** Add about 300 mL of water and a few boiling chips to the second beaker. Place all three test tubes in the beaker, as shown in **Figure B.**
- **6.** Be sure the water level in the beaker is above the tops of the metal samples. Add more water to the beaker if necessary, but do not allow any water to get into the test tubes.
- **7.** Set the beaker of water and test tubes on the hot plate. Turn the hot plate on and heat the water to a boil.
- **8.** While the water is heating, pour about 75 mL of cold water into the graduated cylinder. Measure the volume to the nearest 0.1 mL. Record the volume in the data table column for aluminum.
- **9.** Pour the cold water into the calorimeter. With the top of the calorimeter off, measure the temperature of the water every minute until it stays the same for 3 min. Record this temperature as the initial water temperature in the data table column for aluminum.

- **10.** After the water has been boiling for 10 min, measure the temperature of the boiling water. You can assume that the metal samples are at the same temperature as the water. Record the temperature as the initial metal temperature in the data table columns for each metal. Keep the water boiling.
- 11. Use the test-tube holder to remove the test tube that contains the aluminum sample. CAUTION: Carefully slide the aluminum into the water in the calorimeter without splashing. Quickly put the top on the calorimeter.
- **12.** Insert the thermometer through the hole in the calorimeter top until the tip of the thermometer touches the bottom of the calorimeter.
- **13.** Gently swirl the beaker containing the calorimeter for 30 s while you monitor the temperature. Do not allow the metal sample to hit the thermometer. Record the highest temperature attained by the water as the final temperature in the data table column for aluminum.
- **14.** Remove the aluminum sample from the calorimeter. Pour the water down the drain.
- **15.** Repeat steps 8, 9, and 11–14 for the iron and lead samples.

Hypothesis

Figure B



Cleanup and Disposal

- **1.** Turn off the hot plate. After the boiling water has cooled, pour it down the drain.
- **2.** Dry the metal samples with a paper towel.
- **3.** Make sure your balance is left in the same condition as you found it.
- **4.** Return the metal samples and lab equipment to their proper places.
- **5.** Wash your hands thoroughly with soap or detergent before you leave the lab.

LAB (12)

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

Data and Observations

	Data Table	1	
	Aluminum	Iron	Lead
Mass of metal (g)			
Volume of water (mL)			
Initial water temperature (°C)			
Initial metal temperature (°C)			
Final temperature (°C)			
Change in water temperature (°C)			
Change in metal temperature (°C)			
Heat gained by water (J)			
Specific heat, J/(g·°C)			

- **1.** Calculate the change in water temperature caused by each metal by subtracting the initial water temperature from the final temperature. Record the results in **Data Table 1.**
- **2.** Calculate the change in each metal's temperature by subtracting the final temperature from the initial metal temperature. Record the results in **Data Table 1.**
- **3.** Use the equation in Section 16.1 of your textbook to calculate the amount of heat gained by the water from each metal. (To determine the mass of water, assume the density of water is 1.00 g/mL.) Record the results in **Data Table 1.**
- **4.** Use the same equation to calculate the specific heat of each metal. (Rearrange the equation to solve for specific heat, and assume that the amount of heat lost by the metal equals the amount of heat gained by the water.) Record the results in **Data Table 1.**

Analyze and Conclude

۱.	Applying Concepts To calculate each metal's specific heat, you assumed that the
	amount of heat lost by the metal equals the amount of heat gained by the water. What
	factors determine whether this assumption is valid or not? (Hint: Identify the system and
	the surroundings in this experiment.)

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

Observing and Inferring Which of these metals must release the most heat to
xperience a given decrease in temperature per gram of metal? Explain.
Error Analysis Compare the specific heats you calculated for aluminum, iron, and lead with the values given in Table 16-2 of your textbook. Calculate the percent error if any. Explain possible sources of error in the lab.

Real-World Chemistry

- 1. Would a fishing sinker dropped into an ice-covered lake reach the temperature of the lake water more quickly if the sinker was made of iron or lead? Use your data on the specific heats of these metals to explain your answer. (Assume the sinkers have a starting temperature of 37°C and have the same shape and mass.)
- 2. It is possible to remove a sheet of aluminum foil from a hot oven with your bare hands without burning yourself. However, you will surely burn yourself if you touch a thick aluminum pan in the same oven with your bare hands. Why?
- **3.** One way to identify the composition of metal fragments found at the site of an explosion is to measure the specific heat of the fragments. Suppose a fragment is found to have a specific heat of 0.129 J/(g·°C). Would this information alone be enough to identify the metal in the fragment? Explain why or why not. If not, suggest a method for identifying the metal that would not require any additional equipment.

Energy Changes in Chemical and Physical Processes

hen a chemical or physical process occurs, heat (enthalpy) may be absorbed or released by the system, and the entropy, or disorder, of the system may increase or decrease. Whether a process is spontaneous or not depends upon whether the change in enthalpy of the system ($\Delta H_{\rm system}$) and the change in entropy of the system ($\Delta S_{\rm system}$) are positive or negative. A process is always spontaneous if $\Delta H_{\rm system}$ is negative and $\Delta S_{\rm system}$ is positive, whereas a process is never spontaneous if $\Delta H_{\rm system}$ is positive and $\Delta S_{\rm system}$ is negative. Processes in which $\Delta H_{\rm system}$ and $\Delta S_{\rm system}$ are both positive or both negative are spontaneous only at higher or lower temperatures, respectively. In this activity, you will determine whether some chemical and physical processes are spontaneous.

Problem

How can you determine whether a chemical or physical process is spontaneous?

Objectives

- Measure the temperature of four systems before and after a chemical or physical process.
- Calculate the change in temperature of each system during the process.
- Observe physical changes that occur during each process.
- Deduce whether each process is spontaneous.

Materials

NH₄Cl(s) NaHCO₃(s) 6M HCl 24-well microplate thin-stem pipettes (2) thermometer scissors distilled water toothpicks (1 box)

Safety Precautions



- Always wear safety goggles, gloves, and a lab apron.
- Use extra care when handling all chemicals.
- Notify your teacher of any spills.
- Do not dispose of wastes in the sink or trash can.
- Never place a pipette in your mouth.

Pre-Lab

- **1.** Contrast exothermic and endothermic reactions and processes.
- **2.** Is ΔH_{system} positive or negative in an exothermic process? In an endothermic process?
- **3.** What usually happens to the entropy of a system when a solid or liquid dissolves to form a solution?

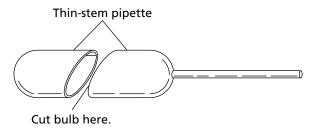
CHEMISTRY SMALL-SCALE LABORATORY MANUAL

- **4.** Read the entire laboratory activity. Write the equation that relates free energy change $(\Delta G_{\rm system})$, $\Delta H_{\rm system}$, $\Delta S_{\rm system}$, and temperature.
- **5.** Is a process spontaneous when ΔG_{system} is positive or negative?

Procedure

- **1.** Use a thermometer to measure the temperature of the air in the room. You may assume that all of the chemicals you will use in this experiment are at room temperature. Record this temperature in **Data Table 1.**
- **2.** Make a chemical microscoop by using scissors to cut off the end of the bulb of a pipette, as shown in **Figure A.**

Figure A



- **3.** Place a half microscoop of solid NH₄Cl in each of wells A1 and B1 on the microplate.
- **4.** Place a half microscoop of solid NaHCO₃ in each of wells A2 and B2 on the microplate.
- **5.** Use the other pipette to add a half pipette of distilled water to well A1.
- **6.** Stir the mixture with a toothpick. Measure the temperature of the mixture. Record the temperature in **Data Table 1.** Also record any physical changes you observe.
- **7.** Rinse the thermometer in cold water.
- **8.** Add a half pipette of distilled water to well A2. Repeat steps 6 and 7.
- **9.** Add a half pipette of 6*M* HCl to well B1. Repeat steps 6 and 7.
- **10.** Add a half pipette of 6*M* HCl to well B2. Repeat steps 6 and 7.

Cleanup and Disposal

- **1.** Dispose of all chemicals and solutions as directed by your teacher.
- **2.** Return all lab equipment to its proper place.
- **3.** Wash your hands thoroughly with soap or detergent before you leave the lab.

Data and Observations

	Data Table 1						
		Tempera	nture (°C)				
Well	Contents	Before mixing	After mixing	Temperature change (°C)	Observations		
A1	NH ₄ Cl + H ₂ O						
A2	NaHCO ₃ + H ₂ O						
B1	NH ₄ Cl + HCl						
B2	NaHCO ₃ + HCl						

Calculate the temperature change in each well by subtracting the temperature before mixing from the temperature after mixing. Record the results in **Data Table 1.**

LAB (13)

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Analyze and Conclude

- **1. Thinking Critically** Why is it valid to assume that all chemicals were at room temperature before they were mixed?
- **2. Classifying** Classify the process that occurred in each well as endothermic or exothermic.
- **3. Applying Concepts** Is ΔH for each process positive or negative?
- **4. Observing and Inferring** Is ΔS for each process positive or negative? (Hint: Think about the physical change that occurred in each process.)
- **5. Drawing a Conclusion** Under what conditions will each process occur spontaneously?

Real-World Chemistry

- **1.** Diamonds are highly valued as gems for jewelry and are also used in industry to make abrasives and cutting tools. Both diamond (S = +2.4 J/K) and graphite (S = +5.7 J/K) are made of pure carbon. Under what conditions, if any, is the conversion of graphite to diamond spontaneous ($\Delta H_{\text{system}} = +1.90 \text{ kJ}$)? Explain.
- **2.** Benzene can be added to gasoline to make an alternative fuel. Is the combustion of benzene spontaneous at 25°C? In the combustion reaction, $\Delta H_{\rm system} = -6535$ kJ and $\Delta S_{\rm system} = -439.1$ J/K.
- 3. Ammonia (NH₃) is used as a refrigerant and as a starting material in the manufacture of fertilizer and explosives. Under the right conditions, solid NH₄Cl decomposes into NH₃ and HCl. In the decomposition reaction, $\Delta H_{\rm system} = +176$ kJ and $\Delta S_{\rm system} = 285$ J/K. What is the lowest temperature at which the reaction will occur spontaneously? (Hint: Assume $\Delta G_{\rm system} = -0.1$ kJ at that temperature.)

CHEMISTRY SMALL-SCALE LABORATORY MANUAL



Determining Reaction Orders

rystal violet is a biological stain that is used in fingerprinting and identifying bacteria. One reason crystal violet is used for these identifications is that it is blue-violet in acidic solutions and colorless in basic solutions. The color change is represented by the chemical equation between the acidic form of crystal violet (CV⁺) and NaOH.

$$C_{25}H_{30}N_3CI + NaOH \rightarrow C_{25}H_{30}N_3OH + NaCI$$

blue-violet (CV⁺) colorless (CV–OH)

Rate is the change in any measurable property per unit of time. Rate is usually expressed as the change in concentration of a reactant per unit of time. The general rate law indicates how the reaction rate is affected by changes in reactant concentrations. Because chemical reactions take place in a series of steps, the reaction rate depends on the slowest step. Therefore, the rate law describes the slowest, or rate-limiting, step in the reaction.

The general rate law for the reaction between CV⁺ and NaOH has the following form in which superscripts are whole numbers that represent the relationship between the rate and the reactant concentration.

Rate =
$$k[CV^+]^n[NaOH]^m$$

If the superscript is equal to zero, the concentration does not affect the reaction rate. A value of 1 means the rate doubles as the concentration doubles, and a value of 2 means the rate quadruples as the concentration doubles. Values for the superscripts must be determined experimentally.

In this activity, you will measure the time needed for the color of CV^+ to disappear as CV-OH is formed. The reaction is complete when the solution becomes colorless. The time and concentration data will be used to calculate the rate of reaction. By determining the rate experimentally with varying concentrations of reactants, the values for m and n in the general rate law can be determined.

Problem

What is the general rate equation for the reaction between crystal violet and sodium hydroxide?

Objectives

- **Measure** the time needed for reactions to occur.
- **Compare** rates of chemical reactions.
- **Infer** general rate equation from experimental data.
- Determine a value for the order of reaction from experimental data.

Materials

droppers (3)

2.0 × 10⁻⁴M crystal violet solution
1.0M NaOH solution
24-well microplate with deep wells

sheets of white paper (15) stopwatch or clock with second hand toothpicks (3) distilled water

LAB (14)

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

Safety Precautions



- Always wear safety goggles, gloves, and a lab apron.
- Crystal violet is moderately toxic and a tissue irritant.
- NaOH is corrosive.
- When water and NaOH are mixed, much heat is released.

Pre-Lab

- **1.** What is the likely value(s) of *m* if the [NaOH] participates in the rate-limiting step of the reaction?
- **2.** How can you tell when the reaction is complete?
- **3.** What is the purpose of placing the well plate on a white sheet of paper?
- **4.** Explain why the value of the average rate is not calculated using the sodium hydroxide concentrations.
- **5.** Read the entire lab activity. Scientists use the balanced chemical equation and previous knowledge of how chemicals react with one another to predict what the rate law might be. Then they run experiments to test their hypotheses. Form a hypothesis as to the values of *m* and *n* in the general rate law for this experiment. Record your hypothesis in the next column.

Procedure

- **1.** Set a 24-well microplate on a sheet of white paper. In three separate wells, mix the water and NaOH in the proportions listed in **Data Table 1.** Use a separate dropper for each liquid. Label the wells by reaction number.
- **2.** Using the third dropper, add the appropriate amount of crystal violet indicated in **Data Table 1** for reaction 1.

- **3.** Check the time (or set the stopwatch) as you add the crystal violet.
- **4.** Stir the mixture with a toothpick. Continue stirring until the blue color disappears and the solution is colorless.
- **5.** As soon as the solution becomes colorless, record the number of seconds it took for the color to disappear in **Data Table 1.**
- **6.** Repeat steps 2 to 5 for reaction mixtures 2 and 3.

Hypothesis	5
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Cleanup and Disposal

- **1.** Wash, dry, and store all glassware.
- **2.** Return all lab equipment to its proper place.
- **3.** Dispose of all solutions as directed by your teacher.
- **4.** Wash your hands thoroughly with soap or detergent before you leave the lab.

Data and Observations

Data Table 1						
Reaction mixture	Crystal violet (drops)	0.1 <i>M</i> NaOH (drops)	Distilled water (drops)	Time (s)		
1	5	20	5			
2	5	10	15			
3	3	20	7			

Data Table 2						
Reaction mixture	Crystal violet (M)	NaOH (<i>M</i>)	Rate (M/s)			
1						
2						
3						

- **1.** To determine the concentrations of crystal violet, multiply the original concentration $(2 \times 10^{-4} M)$ by the number of drops used, and divide by the total number of drops of solution. For reaction #1: $2 \times 10^{-4} M \times 5$ drops/(30 drops solution) = $3.33 \times 10^{-5} M$. Record this value in **Data Table 2.**
- **2.** To determine the concentration of NaOH, multiply the original concentration (1.0M) by the number of drops used, and divide by the total number of drops of solution.
- **3.** Calculate the average rate for each reaction. The acidic form of crystal violet is considered to be zero at the end of the reaction, so the average rate can be calculated from the following equation.

$$\frac{\left[CV^{+}\right]_{final} - \left[CV^{+}\right]_{initial}}{time} = \frac{0 - \left[CV^{+}\right]_{initial}}{time}$$

4. Determine the order of reaction with respect to [CV⁺]. Examine the rate and concentration entries in the calculations table. Reading horizontally, identify the reaction mixtures for which the values for [CV⁺] change but the values for the [NaOH] stay the same. Then substitute the appropriate values into the following ratio. The letters x and z represent the number of the reaction mixture.

$$\frac{\text{Rate}_{x}}{\text{Rate}_{z}} = \frac{k[\text{CV}^{+}]_{x}^{n} [\text{NaOH}]_{x}^{m}}{k[\text{CV}^{+}]_{z}^{n} [\text{NaOH}]_{z}^{m}}$$

The values for the rate constant and for [NaOH] are the same in both reactions, so the ratio reduces to the following.

$$\frac{\text{Rate}_{x}}{\text{Rate}_{z}} = \frac{[\text{CV}^{+}]_{x}^{n}}{[\text{CV}^{+}]_{z}^{n}} = \left[\frac{[\text{CV}^{+}]_{x}}{[\text{CV}^{+}]_{z}}\right]^{n}$$

To find the value of n, the order of reaction with respect to crystal violet, you must take the logarithm of both sides.

$$\log \left[\frac{\text{Rate}_{x}}{\text{Rate}_{z}} \right] = n \log \left[\frac{[\text{CV}^{+}]_{x}}{[\text{CV}^{+}]_{z}} \right]$$

5. Determine the order of reaction with respect to [NaOH]. Examine the rate and concentration entries in the calculations table to identify the reaction mixtures for which the values for [NaOH] change, but the values for the [CV⁺] stay the same. Then substitute the appropriate values into the following ratio and solve for *m*. The letters *x* and *z* represent the number of the reaction mixture.

$$\log \left\lceil \frac{\text{Rate}_x}{\text{Rate}_z} \right\rceil = m \log \left\lceil \frac{[\text{NaOH}]_x}{[\text{NaOH}]_z} \right\rceil$$

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6. Write the general rate law for this reaction, substituting your calculated values for the superscripts m and n.

Analyze and Conclude

1. Thinking Critically A general rate law describes the chemistry of the slowest step in the chemical reaction studied. Acetone is iodinated in the presence of sulfuric acid according to the following chemical equation. How can you account for the absence of [I₂] in the rate law? Rate = [acetone]¹ [HCl]¹

2.	Interpreting Data	Your values for m	n and n may n	not be whole	numbers.	What are	some
	sources of error or in	aprecision with thi	s experiment	t?			

3. Error Analysis Consider that the values for each exponent should be 1. If your values differed, calculate your percent error in determining the values of the two exponents.

Real-World Chemistry

Ethyl acetate reacts with water to form acetic acid and ethanol according to the following reaction. The rate of this reaction is much too slow to be measured, but when HCl is added as a catalyst, the rate quickens significantly. Suppose the rate of reaction doubles when the concentration of ethyl acetate doubles but is unaffected by the amount of water present.

$${
m CH_3C(O)OC_2H_5} + {
m H_2O} \rightarrow {
m CH_3C(O)OH} + {
m C_2H_5OH}$$
 ethyl acetate water acetic acid ethanol

- **a.** Write the general rate law for the uncatalyzed reaction.
- **b.** A catalyst increases the reaction rate by changing the rate-limiting step. Suggest a rate law for the acid-catalyzed reaction.



Observing Equilibrium

nome chemical systems are reversible; that is, they do not go to completion. Instead, they reach a point of equilibrium in which a certain ratio of the concentrations of the products to the concentrations of the reactants is constant. If more reactant particles are added to the system, the equilibrium is disturbed and the system responds by making more products, thus restoring the equilibrium ratio. Similarly, if reactant particles are removed, the products react in the reverse reaction to reform reactant particles and restore equilibrium. If the reactants and products have different colors, shifts in equilibrium can be followed by observing color changes as the system is disturbed.

In this activity, you will observe how the color of the equilibrium mixture of the following chemical system changes when common ions are added and when precipitates are formed.

> Fe³⁺ SCN-FeSCN²⁺ Iron(III) ion thiocyanate ion iron(III) thiocyanate ion

The number of Fe³⁺ ions present in solution will increase if solid Fe(NO₃)₃ is added to the reaction mixture at equilibrium. This means that more FeSCN²⁺ ions will form, and the reaction mixture will have a color strongly resembling the color of the FeSCN²⁺ ion. Similarly, the precipitation of Fe(OH)₃ reduces the number of Fe³⁺ ions in solution, so FeSCN²⁺ ions decompose to form more Fe³⁺ ions and SCN⁻ ions. The color of the solution becomes more like the color of the reactants.

Problem

How do changes in the concentration of reactant ions affect equilibrium?

Objectives

- **Observe** color changes associated with shifts in equilibrium.
- **Relate** changes in reactant concentration to the direction of shift in equilibrium.

Materials

 $0.1M \text{ Fe}(NO_3)_3$ 0.1*M* KSCN 1.0M NaOH 0.1M AgNO₃ $Fe(NO_3)_3$ NH₄SCN KCl distilled water 10-mL graduated cylinder (2) grease pencil droppers (2) medium test tubes (7) stirring rod test-tube rack

Safety Precautions











- Always wear safety goggles, gloves, and a lab apron.
- NH₄SCN produces cyanide fumes when in contact with acids.
- AgNO₃ is toxic by ingestion and will stain skin and clothes.

LAID

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

Pre-Lab

- **1.** How do the concentrations of the ions of a solution change when a double-replacement reaction occurs and a precipitate forms?
- **2.** When a pink solution is mixed with a blue solution, what is the color of the resulting mixture?
- **3.** One milliliter of 0.1*M* HCl is added to the following equilibrium system. Identify the common ion.

$$Co(H_2O)_6^{2+} + 4Cl^- \rightleftharpoons Co(H_2O)_2Cl_4^{2-} + 4H_2O$$

- **4.** Will the equilibrium in question 3 shift toward the reactants (left) or toward the products (right)?
- **5.** Read the entire laboratory activity. Hypothesize the direction of shift in the equilibrium system for this experiment when potassium chloride is added to the reaction mixture. Record your hypothesis in the next column.

Procedure

- **1.** Using a graduated cylinder, measure 4 mL of $0.1M \text{ Fe(NO}_3)_3$ solution into a test tube. Record the color of the Fe³⁺ ion in **Data Table 1.**
- **2.** In a second graduated cylinder, measure 4 mL of 0.1*M* KSCN solution. Record the color of the SCN⁻ ion in **Data Table 1.**
- **3.** Pour the KSCN solution into the $Fe(NO_3)_3$ solution. Record the color of the $FeSCN^{2+}$ ion in **Data Table 1.**
- **4.** Dilute the mixture to about 60 mL with water. Pour 5 mL of this *diluted* solution into each of five test tubes labeled 1–5.

Data and Observations

Data Table 1					
Substance	Color				
Fe(NO ₃) ₃					
KSCN					
FeSCN ²⁺ ion					

- **5.** For each step 6 through 10, record in **Data Table 2** the color of the solution after stirring, whether this color is like the products or like the reactants, the direction of shift in the equilibrium, and the identity of any common ion.
- **6.** To test tube #1, add 0.5 g Fe(NO₃)₃. Stir to dissolve.
- **7.** To test tube #2, add 0.5 g NH₄SCN. Stir to dissolve.
- **8.** To test tube #3, add 0.5 g KCl. Stir to dissolve.
- **9.** To test tube #4, add 3 drops of 1.0*M* NaOH solution. Stir to mix.
- **10.** To test tube #5, add 10 drops of 0.1*M* AgNO₃ solution. Stir to mix.

Hypothesis

Cleanup and Disposal

- **1.** Dispose of all solutions as directed by your teacher.
- **2.** Return all lab equipment to its proper place.
- **3.** Wash your hands thoroughly with soap or detergent before you leave the lab.

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

	Data Table 2						
Test-tube	Substance disturbing	Common		Color is n	ost like	Equilibrium shifts toward	
number	equilibrium	ion added	Color after stirring	Reactants	Products	Left	Right
1	0.5 g Fe(NO ₃) ₃						
2	0.5 g NH ₄ SCN						
3	0.5 g KCl						
4	1.0 <i>M</i> NaOH						
5	0.1 <i>M</i> AgNO ₃						

Analyze and Conclude

- **1. Observing and Inferring** Was a reactant ion or a product ion removed from the equilibrium mixture when you added NaOH and a precipitate formed?
- **2. Observing and Inferring** Based on observations, did the addition of KCl increase or decrease the reactant concentrations? (Hint: When the KCl was added to the equilibrium system, did the equilibrium shift in the same direction as it shifted for the common ion Fe³⁺?)
- **3. Observing and Inferring** Use your data to support or refute the following statement. The precipitate formed after adding AgNO₃ removed SCN⁻ ion rather than FeSCN²⁺ ion.

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

4. Predicting Examine **Data Table 2** and then write a sentence to describe how the equilibrium shifts when

a. reactant ions are removed from solution.

b. reactant ions are added to the solution.

Real-World Chemistry

- 1. Manipulating an equilibrium system to achieve the desired product is a significant part of industrial chemistry. Look up the Haber process for ammonia synthesis and investigate how the conditions of the reaction are manipulated to maximize the amount of ammonia obtained.
- **2.** Bottled carbonated beverages contain carbonic acid that dissolves in water according to the following equation: $CO_2(g) + H_2O(1) \rightleftharpoons H_2CO_3(aq)$. When a bottle is unsealed, the equilibrium is disturbed. What effect does the escaping carbon dioxide gas have on the equilibrium?

CHEMISTRY SMALL-SCALE



Exploring Chemical Equilibrium

eversible chemical reactions reach an equilibrium in which the concentrations of all reactants and products are constant. The relationship between the reactant and product concentrations is defined by the equilibrium constant, $K_{\rm eq}$. In this experiment, you will investigate the reaction in which colorless Fe³⁺ and SCN⁻ ions combine to form red FeSCN²⁺ ions. The intensity of the red color increases with the concentration of FeSCN²⁺. The net ionic equation for this reversible reaction is $Fe^{3+}(aq) + SCN^{-}(aq) \rightleftharpoons FeSCN^{2+}(aq)$.

Problem

Can you calculate the equilibrium constant for a chemical reaction by measuring the concentration of the reaction product?

Objectives

- Prepare serial dilutions of a standard solution.
- Estimate the color intensity of solutions at equilibrium.
- Relate color-intensity values to the concentration of FeSCN²⁺ at equilibrium.
- Calculate the equilibrium constant for the reaction between Fe³⁺ and SCN⁻.

Materials

 $0.200M \text{ Fe}(NO_3)_3$ 0.6M HNO₃ $2.00 \times 10^{-3} M$ KSCN 24-well microplate thin-stem pipettes (6) sheet of white paper

Safety Precautions













- Always wear safety goggles, gloves, and a lab apron.
- Do not operate a pipette with your mouth.
- Use extra care when handling the solutions.
- Do not dispose of materials to be recycled in the sink or trash can.

Pre-Lab

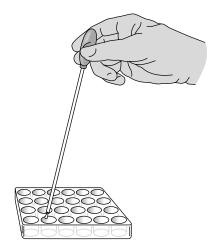
- 1. Contrast homogenous and heterogeneous equilibria. Which type of equilibrium is represented by the reaction between Fe³⁺ and SCN⁻?
- 2. Write the equilibrium constant expression for the reaction between Fe³⁺ and SCN⁻.
- **3.** What effect would decreasing the concentration of Fe³⁺ have on the concentration of FeSCN²⁺ at equilibrium? Explain.
- **4.** What effect would decreasing the concentration of Fe³⁺ have on the equilibrium constant? Explain.
- **5.** Read the entire laboratory activity. Form a hypothesis about how the color intensity will vary in wells A1-A6. Explain your reasoning. Record your hypothesis on page 62.

Procedure

1. Use a pipette to place 10 drops of $Fe(NO_3)_3$ solution in well A1 of the microplate, as shown in Figure A. Record the Fe³⁺ concentration in **Data** Table 1.

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Figure A



- **5.** Repeat step 4 until you have filled all six wells in row A.
- **6.** Add 10 drops of KSCN solution to each well in row A. Carefully stir the contents of each well.
- 7. Place a sheet of white paper beneath the microplate. Estimate the intensity of the red color in each well in row A on a scale from 0 (no red at all) to 1.00 (well A1). Record the intensity values in **Data Table 1.**

Hypothesis

- **2.** Place another 10 drops of Fe(NO₃)₃ solution in well D6. Use a second pipette to add 10 drops of HNO₃ solution to well D6. Stir the mixture carefully with the second pipette.
- **3.** Transfer 10 drops of the mixture in D6 to well A2. Record the Fe³⁺ concentration in the data table. (Hint: The concentration was reduced when you added HNO₃ solution.)
- **4.** Add 10 drops of HNO₃ solution to the mixture that remains in well D6. Stir with a clean pipette. Transfer 10 drops of this mixture to well A3. Record the Fe³⁺ concentration in **Data Table 1.**

Cleanup and Disposal

- **1.** Dispose of all solutions as directed by your teacher.
- **2.** Return all lab equipment to its proper place.
- **3.** Wash your hands thoroughly with soap or detergennt before you leave the lab.

Data and Observations

	Data Table 1							
Well	[Fe ³⁺] before mixing (<i>M</i>)	[Fe ³⁺] after mixing (<i>M</i>)	[SCN ⁻] after mixing (<i>M</i>)	Color intensity	[FeSCN ²⁺] at equilibrium (<i>M</i>)	[SCN ⁻] at equilibrium (<i>M</i>)	[Fe ³⁺] at equilibrium (<i>M</i>)	К _{еq}
A1								_
A2								
A3								
A4								
A5								
A6								

LAB (16)

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

- **1.** Calculate the concentration of Fe³⁺ in each well after mixing with KSCN. (Hint: Remember that 10 drops of KSCN solution was added to 10 drops of Fe(NO₃)₃ solution in each well.) Record the results in **Data Table 1.**
- **2.** Calculate the concentration of SCN⁻ in each well after mixing. (Hint: The value will be the same for all wells.) Record the results in **Data Table 1.**
- **3.** Calculate the concentrations of FeSCN²⁺, SCN⁻, and Fe³⁺ in well A1 at equilibrium. To make these calculations, assume that all of the SCN⁻ was consumed in the reaction in this well. Record the results in **Data Table 1.**
- **4.** Calculate the concentration of FeSCN²⁺ in wells A2–A6 at equilibrium. Multiply the equilibrium concentration in well A1 by the color intensity in each well. Record the results in **Data Table 1.**
- **5.** Calculate the concentration of SCN⁻ in wells A2–A6 at equilibrium. Subtract the concentration of FeSCN²⁺ at equilibrium from the concentration of SCN⁻ after mixing. Record the results in **Data Table 1.**
- **6.** Calculate the concentration of Fe³⁺ in wells A2–A6 at equilibrium. Subtract the concentration of FeSCN²⁺ at equilibrium from the concentration of Fe³⁺ after mixing. Record the results in **Data Table 1.**
- **7.** Calculate the equilibrium constant, $K_{\rm eq}$, using the equilibrium concentrations of FeSCN²⁺, SCN⁻, and Fe³⁺ in wells A2–A6. Record the results in **Data Table 1.**

Analyze and Conclude

Drawing a Conclusion Did your results support your hypothesis? Explain.
Applying Concepts How would the results differ if the experiment was done at a higher temperature?
Collecting and Interpreting Data Based on the values you calculated for $K_{\rm eq}$, do you think the procedure was precise? Explain why or why not. (Hint: Values that are within the same order of magnitude may be considered precise.)
same order of magnitude may be considered precise.)

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

4. Error Analysis Explain possible sources of imprecision in the experiment.

Real-World Chemistry

- **1.** Many homes have water pipes made of iron, which can slowly release Fe³⁺ ions into the water. Although these ions are not a health hazard, at high concentrations they can discolor dishes and laundry. Explain how KSCN could be used in a home test kit for measuring the level of Fe³⁺ in water.
- **2.** Acetic acid (CH₃COOH) and ethanol (C₂H₅OH) react to form ethyl acetate (CH₃COOC₂H₅) which is used to make artificial silk. Water is also produced in this reaction.

 $CH_3COOH + C_2H_5OH \rightleftharpoons CH_3COOC_2H_5 + H_2O$ Explain why it is important to remove ethyl acetate as it forms. What other steps could be taken to increase the production of ethyl acetate?

17

CHEMISTRY SMALL-SCALE LABORATORY MANUAL



Comparing the Strengths of Acids

cids ionize and produce hydrogen ions in aqueous solution. The strength of an acid depends on how completely it ionizes. Strong acids undergo essentially complete ionization, whereas weak acids are only partly ionized at equilibrium. Like other reversible reactions, the ionization of a weak acid can be represented by an equilibrium constant expression. The value of this expression, called the acid ionization constant, K_a , is a measure of the strength of a weak acid.

Problem

Can you compare the strengths of several acids by measuring the pH of their solutions and calculating K_a ?

Objectives

- Prepare serial dilutions of standard acid solutions.
- Measure the pH of the standard and diluted solutions.
- Calculate the H⁺ ion concentration of each solution and the K_a of each weak acid.
- Rank the acids in order of strength.

Materials

0.100*M* boric acid (H₃BO₃) 0.100*M* citric acid (H₃C₆H₅O₇) 0.100*M* hydrogen peroxide (H₂O₂) 0.100*M* permanganic acid (HMnO₄) 24-well microplate thin-stem pipettes (12) pH papers (various pH ranges) distilled water

Safety Precautions



- Always wear safety goggles, gloves, and a lab apron.
- Use extra care when handling the solutions.
- Notify your teacher of any chemical spills.
- Do not dispose of materials to be recycled in the sink or trash can.

Pre-Lab

- **1.** Explain the difference between the terms *weak acid* and *dilute acid*.
- **2.** Formic acid has a K_a of 1.8×10^{-4} . Acetic acid has a K_a of 1.8×10^{-5} . Which acid is stronger? Explain your answer.
- **3.** Define pH.
- **4.** Read the entire laboratory activity. Form a hypothesis about the effect of decreasing the concentration of a weak acid on the pH of the acid solution and on the K_a of the acid. Record your hypothesis on page 66.
- **5.** Write the equilibrium constant expression for this reaction: $HA(aq) \rightleftharpoons H^+(aq) + A^-(aq)$.

Procedure

- **1.** Use a pipette to place 18 drops of distilled water in each well in columns 2 and 3 of the microplate.
- **2.** Place about 20 drops of 0.100*M* boric acid in well A1. Record the name of the acid and the concentration in **Data Table 1.**
- **3.** Transfer 2 drops of the solution in well A1 to the distilled water in well A2. Carefully stir the mixture in well A2 with the pipette.
- **4.** Transfer 2 drops of the solution in well A2 to the distilled water in well A3. Carefully stir the mixture in well A3 with the pipette.

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

- **5.** Repeat steps 2–4 with citric acid, hydrogen peroxide, and permanganic acid. Place each acid in different rows of the microplate.
- **6.** Using pH paper, measure the pH of the solutions in wells A1–D1 and A3–D3. If a paper fails to indicate a pH value for a well, try a paper with a different pH range. Record the pH values in **Data Table 1.**

Hypothesis		

Cleanup and Disposal

- **1.** Dispose of all solutions as directed by your teacher.
- **2.** Return all lab equipment to its proper place.
- **3.** Wash your hands thoroughly with soap or detergent before you leave the lab.

Data and Observations

	Data Table 1								
Well	Acid	Initial [Acid] (<i>M</i>)	рН	[H ⁺] at equilibrium (<i>M</i>)	[Acid] at equilibrium (<i>M</i>)	K _a	Average <i>K</i> _a		
A1									
A3									
B1									
В3									
C1									
C3									
D1									
D3									

- **1.** Calculate the initial concentration of acid in wells A3–D3. (Hint: Remember that the solutions in each of these wells were made by two 10-fold dilutions of the solutions in wells A1–D1.) Record your results in **Data Table 1.**
- **2.** Calculate the concentration of H⁺ ions in wells A1–D1 and A3–D3 at equilibrium. (Hint: Use the formula that relates pH and [H⁺].) Record your results.
- **3.** Calculate the concentration of acid in wells A1–D1 and A3–D3 at equilibrium. (Hint: Subtract the concentration of H⁺ ions at equilibrium from the initial concentration of acid.) Record your results in **Data Table 1.**
- **4.** Calculate the acid ionization constant, K_a , for each acid, using the equilibrium concentrations of acid and H⁺ in wells A1–D1 and A3–D3. (Hint: Remember that the concentration of the conjugate base, A⁻, equals the concentration of H⁺.) Record your results. If the concentration of acid at equilibrium is zero in any well, draw a dash in the K_a column for that cell.
- **5.** Calculate the average K_a for each acid. Record your results in **Data Table 1.**

SMALL-SCALE LABORATORY MANUAL

Analyze and Conclude

- **1. Applying Concepts** If the concentration of acid at equilibrium is zero in any well, you can conclude that the acid in that well is a strong acid. Explain why.
- **2. Drawing a Conclusion** Rank the acids in order of strength based on your results. Indicate whether each acid is a strong or weak acid.
- **3. Error Analysis** Look up the accepted value of K_a for each weak acid. Compare the average K_a you calculated for each acid with the accepted value. Calculate the percent error if any. Explain possible sources of error in the lab.

Real-World Chemistry

- 1. Your body contains a large number of acids, which serve a wide variety of functions. Included are 20 different kinds of amino acids, which are linked to form proteins and the nucleic acid DNA, which stores genetic information. Look up the acid ionization constants of amino acids and DNA. How do the strengths of these acids compare with those of the acids you studied in this activity?
- **2.** The hydrangea is a popular garden shrub that produces large, round clusters of flowers. Some hydrangeas produce blue flowers when they grow in acidic soil and pink flowers when they grow in basic soil. Would it be a good idea for
- a gardener to fertilize a hydrangea with compost made from lemon and orange rinds if she wanted the plant to produce pink flowers? Explain your answer.
- 3. Acid rain is rainwater that has a pH lower than the normal value of about 5.5. It has been blamed for the corrosion of buildings and statues and for widespread environmental damage. Government regulations aimed at reducing acid rain focus mainly on limiting the release of sulfur oxides and nitrogen oxides into the air. Explain why. (Hint: Recall what you have learned about anhydrides.)



CHEMISTRY SMALL-SCALE LABORATORY MANUAL



Testing the Acidity of Aspirin

spirin is a medicine that has been used for over a century to relieve pain, reduce fever, and fight inflammation. Aspirin is the common name for acetylsalicylic acid ($C_9H_8O_4$). Because aspirin is an acid, it can cause an upset stomach in some people who use it. Therefore, many drug companies also produce buffered aspirin, a pain reliever that is designed to be gentler on the stomach.

Problem

How can you use a base to test the difference between buffered and unbuffered aspirin?

Objectives

- Measure the pH of solutions of unbuffered and buffered aspirin.
- **Titrate** each solution with a base.
- Make and use a graph of pH versus volume of base for the two pain relievers.

Materials

0.1*M* NaOH unbuffered aspirin tablet buffered aspirin tablet 10-mL graduated cylinder 50-mL beaker mortar and pestle thin-stem pipettes (3) stirring rod pH paper distilled water plastic spoon

Safety Precautions



- Always wear safety goggles, gloves, and a lab apron.
- Use extra care when handling the solutions.
- Notify your teacher of any chemical spills.
- Do not dispose of materials to be recycled in the sink or trash can.

Pre-Lab

- **1.** What happens in a neutralization reaction?
- **2.** What is a buffer?
- **3.** Suppose a buffer contains 0.1 mole of a weak acid (HA) dissolved in water. What else must the buffer contain?
- **4.** How can you identify the equivalence point of a titration by examining a graph of pH versus volume of base added?
- **5.** Read the entire laboratory activity. Form a hypothesis about which pain reliever will show a smaller change in pH when base is added. Explain why. Record your hypothesis on page 70.

Procedure

- **1.** Measure 5.0 mL of distilled water in a 10-mL graduated cylinder. Remember to take the volume reading at the bottom of the meniscus.
- 2. Fill a pipette with distilled water. Count the number of drops that must be added from the pipette to bring the water level in the graduated cylinder to the 6.0-mL mark. Record this number in **Data Table 1.**
- **3.** Using a mortar and pestle, grind one tablet of unbuffered aspirin into a fine powder. Transfer the ground tablet to a 50-mL beaker.
- **4.** Add 20 mL of distilled water to the beaker. Using a stirring rod, stir the mixture until the powder is dissolved.

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- **5.** Measure the pH of the solution with pH paper. Record the pH in **Data Table 1.**
- **6.** Use the pipette to add 1.0 mL of 0.1*M* NaOH to the beaker. Stir the mixture.
- **7.** Measure the pH of the solution with pH paper. Record the pH in the data table column for each additional 1.0 mL of NaOH added.
- **8.** Repeat steps 6 and 7 until you have added 5.0 mL of NaOH.
- **9.** Repeat steps 3–8 using one tablet of buffered aspirin.
- **10.** Calculate the change in pH for each pain reliever by subtracting the pH before adding NaOH from the pH after adding 5.0 mL of NaOH. Record your results in **Data Table 1.**

Hypotnesis		

Cleanup and Disposal

- **1.** Dispose of all solutions as directed by your teacher.
- **2.** Return all lab equipment to its proper place.
- **3.** Wash your hands thoroughly with soap or detergent before you leave the lab.

Data and Observations

Data Table 1							
Number of drops in 1.0 mL:							
pH before pH after adding NaOH							
Pain reliever	NaOH	1.0 mL	2.0 mL	3.0 mL	4.0 mL	5.0 mL	Change in pH
Unbuffered aspirin							
Buffered aspirin							

Analyze and Conclude

1. Making and Using Graphs Make a graph of pH versus volume of NaOH added. Plot the data for both pain relievers on the same graph. Draw lines between the data points for each curve. Label both curves and both axes, and give the graph a title.

Making and Using Graphs Does your graph indicate that either pain reliever was titrated to its equivalence point? Explain your answer.
 Collecting and Interpreting Data Which pain reliever is more acidic?
 Error Analysis Compare the results of this experiment with your hypothesis. Explain possible reasons for any disagreement.

Real-World Chemistry

- 1. Scientists have discovered the remains of dead organisms in peat bogs that are well preserved after hundreds or even thousands of years. Tannic acid, a compound found in peat bogs, slows the breakdown of the dead organisms' tissues, although it usually destroys the DNA in the tissues. However, the DNA is protected from destruction if the area contains limestone, a mineral made of calcium carbonate (CaCO₃). Explain how calcium carbonate protects DNA. (Hint: Refer to **Table 19-2** in your textbook.)
- 2. Methanoic acid (HCOOH) is used in the processing of leather. To determine the concentration of methanoic acid in the process residue, chemists can titrate the residue with a base. Suppose a 75.0-mL sample of residue is neutralized by 42.0 mL of 0.200*M* NaOH. What is the molarity of methanoic acid in the residue? (Assume the residue contains nothing but methanoic acid and water.)
- **3.** Solutions sold for cleaning and storing contact lenses are usually buffered. Why do you think that is so?

CHEMISTRY SMALL-SCALE LABORATORY MANUAL



Reduction of Manganese

transfer of electrons between reactants identifies oxidation–reduction reactions. A reducing agent releases electrons that are acquired by the oxidizing agent. Permanganate ions are excellent oxidizing agents because they contain manganese atoms (Mn) with a high oxidation number. The intense color of these ions acts as an indicator that marks the end of the redox reaction in much the same way that phenolphthalein marks the end of an acid–base reaction. But, because permanganate ions decompose easily, $KMnO_4$ solutions have to be titrated against a primary standard shortly before use to get their exact concentrations. A primary standard has a known concentration. For $KMnO_4$ solutions, the primary standard is sodium oxalate, $Na_2C_2O_4$, in an aqueous solution of sulfuric acid.

In this activity, a measured amount of $Na_2C_2O_4$ is dissolved in H_2SO_4 according to the following equation:

$$H_2SO_4(aq) + Na_2C_2O_4(s) \rightarrow Na_2SO_4(aq) + H_2C_2O_4(aq)$$

This colorless solution is heated, and $KMnO_4$ is added dropwise until the solution turns faint pink. The permanganate ions react with the oxalate ions according to the following reaction:

$$\begin{array}{c} {\rm 5H_2C_2O_4(aq)} \, + \, 2{\rm KMnO_4(aq)} \, + \, 3{\rm H_2SO_4(aq)} \, \to \\ {\rm 10CO_2(g)} \, + \, 8{\rm H_2O(I)} \, + \, 2{\rm MnSO_4(aq)} \, + \, {\rm K_2SO_4(aq)}. \end{array}$$

From the mass of the $\mathrm{Na_2C_2O_4}$, the volume of potassium permanganate solution, and the stoichiometry of the balanced chemical equation, the concentration of $\mathrm{KMnO_4}$ can be determined.

Problem

How can a standard solution containing a reducing agent be used to find the unknown concentration of an oxidizing agent?

Objectives

- Calculate a value for the molarity of the KMnO₄ solution using stoichiometry and volume data.
- Write and balance oxidation-reduction equations.

Materials

KMnO₄ solution 1.0*M* solution of H₂SO₄ 0.1 g Na₂C₂O₄ 100-mL beaker 10-mL graduated cylinder micropipettes (2) test tubes (3) thermometer test-tube holder test-tube rack hot plate grease pencil stirring rod distilled water

Safety Precautions



- Always wear safety glasses, gloves, and a lab apron.
- Acids can be corrosive. Handle with care.
- Sodium oxalate is toxic and an irritant.
- Potassium permanganate is toxic and an irritant.
- Never place a pipette in your mouth.

LAB (19

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

Pre-Lab

- **1.** Write and balance the net ionic equation for the redox reaction between $KMnO_4$ and $H_2C_2O_4$.
- **2.** What is the oxidation state of manganese in MnO_4^- ? In $MnSO_4$?
- **3.** According to the balanced equation, how many electrons are transferred?
- **4.** Suppose that 30.00 mL of KMnO₄ solution exactly reacts with 0.250 g of Na₂C₂O₄ that has been dissolved in H₂SO₄. Calculate the molarity of the KMnO₄ solution.
- **5.** Read the entire laboratory activity. If the KMnO₄ solution is exactly 0.04*M*, how many drops will be needed to react with 0.10 g Na₂C₂O₄? (Hint: There are 24 drops in 1 milliliter.)
- **6.** Hypothesize about what element in the KMnO₄ and NaC₂O₄ is oxidized and what element is reduced. Record your hypothesis below.

Procedure

- **1.** Fill a 100-mL beaker with distilled water. Fill one micropipette with distilled water. Count the number of drops needed to fill a 10-mL graduated cylinder to the 1.00 mL mark. Record the number of drops in **Data Table 1.** Repeat this process two more times. Label this pipette "KMnO₄."
- **2.** Repeat step 1 for the second micropipette. Label this pipette "H₂SO₄."
- 3. Prepare a hot-water bath. Adjust the water level in the 100-mL beaker to 75 mL. Set the beaker on a hot plate and heat the water to between 80°C and 90°C. Do not boil the water. This causes the formation of a murky brown solution, and the endpoint cannot be reached.
- **4.** Measure and record the mass of 0.10 g Na₂C₂O₄ to the nearest hundredth of a gram. Transfer the solid to a test tube, and set the test tube in a test-tube rack. Label a second small test tube "WASTE" and set it in the test-tube rack.
- **5.** Rinse the calibrated H₂SO₄ micropipette with a small amount of the sulfuric acid solution. Put the H₂SO₄ rinse into the waste test tube. Partially fill the micropipette with 1.0*M* sulfuric

- acid solution, and add 2 mL of this acid to the test tube containing the $Na_2C_2O_4$.
- **6.** Using a test-tube holder, place the test tube containing the acidic oxalate solution into the water bath. Allow the solution to warm for 5 minutes.
- 7. While the test tube is warming, rinse the 10-mL graduated cylinder with KMnO₄. Put the KMnO₄ rinse into the waste test tube. Then fill the graduated cylinder with 10 mL of the KMnO₄ solution.
- **8.** Fill a third test tube halfway with distilled water. Rinse the KMnO₄ micropipette with a small amount of the potassium permanganate solution. Put the KMnO₄ rinse into the waste test tube. Partially fill the micropipette with KMnO₄ solution from the graduated cylinder, and add 1 drop to the test tube containing distilled water. This is your titration standard; it is the color of the reaction mixture at the endpoint.
- **9.** Using the KMnO₄ micropipette, add 3 drops of the KMnO₄ solution into the test tube warming in the water bath. Keep the test tube in the water bath. Stir until the color disappears (5 minutes or less).
- **10.** After the color disappears, keep the temperature of the water bath above 65°C and continue to add drops of KMnO₄ one at a time while stirring constantly. Add drops of KMnO₄ until the added drop turns the solution the same pink color as the titration standard. The color should persist for 30 s. Record the total number of drops in **Data Table 2.**

Hypothesis

Cleanup and Disposal

- **1.** Dispose of all chemicals as directed by your teacher.
- 2. Return all lab equipment to its proper place.
- **3.** Wash your hands thoroughly with soap or detergent before you leave the lab.

LAB (19)

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Data and Observations

Data Table 1						
	Number of drops in 1 mL					
Micropipette used for:	Trial 1	Trial 2	Trial 3	Average		
KMnO ₄						
H ₂ SO ₄						

Data Table 2	
Mass of sodium oxalate (g)	
Drops of KMnO ₄ needed to reach endpoint	
Volume of KMnO ₄ needed to reach endpoint (mL)	

- **1.** Calculate the average number of drops in 1 milliliter for your micropipettes. Record this number in **Data Table 1.**
- 2. Multiply the drops of KMnO₄ needed to reach endpoint by the average number of drops per milliliter to calculate the volume of the KMnO₄ solution used. Record this number in Data Table 2.
- 3. Calculate the molar mass of sodium oxalate and record the mass in Data Table 2.
- **4.** Calculate the number of moles of sodium oxalate.
- **5.** Using the balanced chemical equation and the number of moles of the $Na_2C_2O_4$, calculate the number of moles of $KMnO_4$ used.
- **6.** Divide the number of moles of KMnO₄ (from question 5) by the volume of KMnO₄ (from question 2). This is the exact molarity, the standardized concentration.

Analyze and Conclude

- **1. Observing and Inferring** What is the purpose of calibrating the micropipette?
- 2. **Observing and Inferring** What color was the solution containing H₂SO₄ and Na₂C₂O₄? Containing KMnO₄? Use the balanced net ionic equation to infer why the pink color occurs at the endpoint.

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3. Error Analysis Explain possible sources of error in this activity.

Real-World Chemistry

- 1. Chlorine is used as a bleach and disinfectant, but totally dry chlorine has no bleaching power. Chlorine owes its bleaching capability to the hypochlorous acid, HClO, that is formed when chlorine reacts with water. Once the HClO is formed, it readily gives up oxygen, O, to any nearby oxidizable substance. The following chemical equations describe these two reactions:
 - **a.** $Cl_2 + H_2O \rightarrow HCl + HClO$
 - **b.** $HClO \rightarrow HCl + O$

Would you expect the bleaching action of HClO to be the result of its activity as an oxidizing agent or a reducing agent? Explain your reasoning.

2. Manganese dioxide is a decomposition product of the permanganate ion. It is also part of the cathode in an alkaline flashlight battery. Examine the cathodic half-reaction:
2MnO₂(s) + H₂O(l) → Mn₂O₃(s) + 2OH⁻(aq) Is MnO₂ an oxidizing agent or a reducing agent? Which process takes place at the cathode—oxidation or reduction?

20 CHEMISTRY SMALL-SCALE LABORATORY MANUAL



Plants Produce Oxygen

n ecosystem is the sum total of all organisms and their environments within a given area. For most ecosystems, sunlight is the ultimate energy source. Green plants, algae, and some bacteria absorb the energy from the red, blue, and indigo wavelengths of the sunlight. The light energy facilitates the production of oxygen and carbohydrates from carbon dioxide and water.

$$6CO_2 + 6H_2O \xrightarrow{\text{light energy}} C_6H_{12}O_6 + 6O_2$$

$$\xrightarrow{\text{chlorophyll}} enzymes$$

This conversion process, photosynthesis, was observed by Priestley in 1772. He observed that when a plant and an animal were kept together in an airtight jar, both lived; but when kept separately, both died. Later, investigators realized that the plants produce oxygen, while the animal exhales carbon dioxide and water vapor. To be complete, this miniecosystem requires complimentary processes.

Researchers discovered that the energy needed for photosynthesis was obtained from the red and blue wavelengths of light absorbed by green chloroplasts in plant leaves. Oxygen production in the chloroplasts of algae then led scientists to hypothesize that photosynthesis took place in the chloroplasts. Because oxygen gas is a product of photosynthesis, monitoring its production is an indirect measure of the amount of photosynthesis.

In this activity, you will prepare two test samples containing an indicator and different amounts of CO_2 . As CO_2 is consumed to produce oxygen, the color of the solution changes. By observing how quickly the color change occurs, you can qualitatively evaluate the dependence of photosynthesis on carbon dioxide concentration. Because oxygen gas is colorless and odorless at room temperature and pressure, methylene blue is used as an indicator. Oxygen gas is the only gas that removes the color from methylene blue.

Problem

How does carbon dioxide concentration affect the rate of photosynthesis?

Objectives

- **Observe** the production of oxygen gas using an indicator.
- Relate the rate of oxygen production to carbon dioxide concentration.
- **Infer** the effect of carbon dioxide concentration on photosynthesis.

Materials

0.2% sodium
hydrogen carbonate (NaHCO₃)
solution
methylene blue
indicator solution
green leaves
plastic weighing
cup

micropipettes (2) small test tubes (3) scissors scoop or equivalent stirring rod test-tube rack distilled water light source

SMALL-SCALE LABORATORY

Safety Precautions









- Always wear safety goggles, gloves, and a lab apron.
- Methylene blue will stain skin and clothing.
- Never place the pipette in your mouth.

Pre-Lab

- **1.** Read the entire laboratory activity. Sodium hydrogen carbonate dissolved in water is a source of CO₂. The chemical equation for photosynthesis shows the relationship between CO_2 and O_2 . Form a hypothesis as to which substance— NaHCO₃ or distilled water—will cause photosynthesis to occur more rapidly. Record your hypothesis in the next column.
- **2.** Read a biology book to learn what chloroplasts are and where they are found. What is their function?
- **3.** What is the purpose of a test tube with distilled water but no leaf pieces?
- **4.** When methylene blue loses its color, what gas is present? Explain.

Procedure

- 1. Using a micropipette, fill two small test tubes half full with distilled water and set the tubes in a testtube rack. Fill a third small test tube half full with 0.2% sodium hydrogen carbonate solution and set it in the test-tube rack. Add 3 drops of methylene blue solution to each test tube.
- 2. Using the scissors, cut a green leaf into pieces small enough to fit into the test tubes. Collect the pieces in the plastic weighing cup.
- **3.** Using a scoop, divide the leaf pieces between one of the test tubes filled with distilled water and the test tube filled with sodium hydrogen carbonate solution. Be sure to add the same quantity of leaves to each test tube.

- **4.** Using a stirring rod, mix the leaves into each solution. (Leaves may remain near the surface of the liquid.) Then place the three test tubes in a sunny window or under a lamp.
- **5.** Record your observations of the solutions in the 0 min column of **Data Table 1.**
- **6.** Record your observations of the solutions at 1-min intervals until the solution decolorizes. (More entries may be needed if the day is cloudy.)

Hypothesis

Cleanup and Disposal

- **1.** Dispose of all chemicals as directed by your teacher.
- **2.** Return all lab equipment to its proper place.
- **3.** Wash your hands thoroughly with soap or detergent before you leave the lab.

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

Data and Observations

Data Table 1							
	Time elapsed (min)						
Solution	0	1	2	3	4	5	6
Distilled water + methylene blue							
Distilled water + leaf pieces + methylene blue							
Na ₂ HCO ₃ solution + leaf pieces + methylene blue							

Analyze and Conclude

1. Observing and Inferring Which test tube better supports photosynthesis? Support your answer with evidence from your observations.

2. Drawing a Conclusion What is the relationship between CO_2 concentration and the rate of photosynthesis?

3. Measuring and Using Numbers Starch is the storage carbohydrate in plants. Starch is a polymer of glucose molecules that were formed during photosynthesis. Suppose 5 mL of O_2 gas is produced during a photosynthesis experiment similar to this one. If the gas was collected at 22.0°C and a barometric pressure of 754 mm Hg, what mass of glucose $(C_6H_{12}O_6)$ was produced? (Hint: PV = nRT; replace the general carbohydrate formula in the photosynthesis equation with the formula for glucose.)

CHEMISTRY SMALL-SCALE LABORATORY MANUAL

4. Designing an Experiment/Identifying Variables How could the experimental design be modified to collect and measure the amount of O₂ gas produced?
5. Error Analysis Explain possible sources of error in this activity.

Real-World Chemistry

- **1.** Greenbelts are narrow areas of trees, shrubs, and other greenery that are planted around a community. Automobiles are propelled by the combustion of fossil fuels. Why is it important to have greenbelts along major freeways?
- **2.** Oxygen promotes deterioration of food products. One patented indicator for detecting oxygen in food packaging includes methylene blue in its formulation. For what reason would methylene blue be included? What advantage might it have over other indicators?

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